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RESEARCH ARTICLE

REMOVAL OF NUTRIENT SALTS: COMPARATIVE STUDY OF BIOSORBENTS DERIVED FROM COCOA AND RUBBER AGRICULTURAL WASTES

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ABSTRACT

Within the framework of sustainable development and the circular economy, it is essential to utilize natural materials derived from agricultural waste as adsorbents. The objective of this study is to investigate the removal capacities of nitrates and phosphates using biosorbents derived from cocoa shells and rubber tree residues, in order to identify the most suitable biosorbent for each type of pollutant.The optimal conditions for biosorbent preparation were determined using experimental design methodology. Throughout the study, including the Plackett–Burman design (PBD), the removal efficiency of nitrates and phosphates was used as the sole response indicator to evaluate material performance.The results showed that cocoa shells were more effective for phosphate removal, whereas rubber-based materials exhibited better performance for nitrate adsorption. This difference is attributed to the physicochemical properties of the ions and the surface characteristics of the biosorbents.

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INTRODUCTION

Water resource pollution by nutrients, particularly nitrates (NO_3^-) and phosphates (PO_4^{3-}), represents a major cause of aquatic ecosystem degradation worldwide (Carpenter *et al*., 1998; Smith *et al*., 1999). These nutrients mainly originate from intensive agricultural practices, domestic and industrial wastewater discharges, and runoff from fertilized soils (Withers & Jarvie, 2008). Excessive enrichment of aquatic environments in nitrates and phosphates leads to eutrophication, characterized by abnormal proliferation of algae and macrophytes. This process results in decreased dissolved oxygen, deterioration of water quality, and, in extreme cases, the death of aquatic organisms (Conley *et al*., 2009). Phosphates are often identified as the primary limiting factor in freshwater eutrophication, whereas nitrates play a key role in marine and estuarine systems (Conley *et al*., 2009). Moreover, high nitrate concentrations in drinking water pose health risks, particularly infant methemoglobinemia, which has led to the establishment of strict standards by the World Health Organization (WHO, 2017). Several conventional methods exist for nitrate and phosphate removal (Bhatnagar & Sillanpää, 2011). However, in the context of sustainable development, there is a growing need to use natural, low-cost materials derived from agricultural waste as adsorbents. These materials are rich in functional groups favorable for ion adsorption (Crini, 2006). Numerous studies have demonstrated the effectiveness of plant-based biosorbents in removing nitrates and phosphates from aqueous solutions (Foo & Hameed, 2010; Loganathan *et al*., 2014). Cocoa shells and rubber residues are particularly promising due to their lignocellulosic composition (cellulose, hemicellulose, and lignin), which enhances interactions with dissolved nutrients (Ahmad *et al*., 2014). Thus, this study aims to evaluate the removal efficiency of nitrates and phosphates using biosorbents derived from cocoa shells and rubber residues, in order to identify the most suitable biosorbent for each pollutant and to develop an optimized biosorbent for water treatment.

MATERIALS AND METHODS

Biosorbent Preparation

Biomass collection and pretreatment: Cocoa shells and rubber residues were collected from producers in Daloa (western Côte d'Ivoire). The materials were washed with tap water to remove impurities. Biosorbent preparation involved four main steps: drying, grinding, sieving, and washing.

Drying: Cocoa shells were air-dried, considering that rubber shells naturally dry on trees before falling.

Grinding: The dried biomass was ground to obtain homogeneous particles. This was done in two stages: manual grinding followed by mechanical grinding using a Retsch SK 100 knife mill.

Sieving: Ground particles were separated using three sieves with different mesh sizes to ensure uniformity and reproducibility in biosorption tests.

Washing: The material was treated with NaOH or H_2SO_4 solutions at predefined concentrations based on the experimental design. The suspension was stirred at 350 rpm using a magnetic stirrer (HI 200M) under specified time and temperature conditions. The material was then rinsed with tap water, followed by distilled water, and finally dried.

Optimization using experimental design: The optimal conditions for biosorbent preparation were determined using the experimental design methodology, which contrasts with the "one-factor-at-a-time" approach that consists of varying each factor sequentially while assigning all possible values (Tinsson, 2010). Throughout the study, the phosphate removal efficiency was used as the sole response to evaluate the performance of the material. First, a Plackett–Burman design (PBD) was employed to identify the factors likely to influence the response. The investigated factors included the nature of the biomass (X1), particle size (X2), activation time (X3), temperature (X4), mass ratio (X5), type of activating agent (X6), and normality (X7). In this type of design, each factor is studied at two levels: a low level (−) and a high level (+). These levels correspond to numerical values for quantitative factors and to defined states for qualitative factors. Table 1 below presents the coded and actual values of the factors, as well as the experimental domain used in this design.

Table 1. Factors and experimental domain for the Plackett–Burman design

Coded variables (X_i)	Factors (U_i)	Experimental domain	
		Low level (−1)	High level (+1)
X1	U_1 : Nature of the biomass	Cocoa	Rubber
X2	U_2 : Particle size (µm)	A	B
X3	U_3 :Activation time (h)	2	4
X4	U_4 : Temperature (°C)	30	60
X5	U_5 : Mass ratio	10	20
X6	U_6 : Type of activating agent	H_2SO_4	NaOH
X7	U_7 : Normality (N)	0,1	0,5

In this table, A represents particle sizes ranging from 0.2 mm to 0.5 mm, while B corresponds to particle sizes between 0.5 mm and 1 mm. The mathematical model relating the response ER (removal efficiency) to the factors Xi is a first-order model without interactions, expressed by the following equation (Eqn. 1):

$$E_R = a_0 + \sum_{i=1}^{7} \text{aiXi}$$

In this relationship, a_0 represents the constant (or mean effect) of the model, while ai corresponds to the effects of the factors. These effects make it possible to determine whether a given factor Xi has a significant influence or not.

Model validation and statistical analysis: Several criteria exist to assess the validity of models and their experimental design coefficients. In this study, factor coefficients and model significance were evaluated using multiple regression analysis and ANOVA. Model terms were retained or eliminated based on the p-value, with a confidence level of 95% ($p < 0.05$). Model significance was first assessed by comparing the mean square of the regression with the mean square of the residuals. According to Bezerra et al. (2019), a model fit is considered adequate when the sum of squares due to residuals (errors) is less than one-third of the sum of squares due to the model. In addition, the sum of squares due to pure error was compared with the total sum of squares Tinsson (Tinsson, 2010). The quality of the models was also evaluated using the coefficients of determination R^2 and adjusted R^2, and their statistical significance was verified using Fisher's F-test Maataoui (Maataoui et al., 2025). Finally, these results were confirmed by analyzing the coefficient of variation (CV) given by the equation below (Eqn. 2):

$$C_V = \frac{1}{n}\left(\sum_{i=1}^{n} \frac{\text{ERexp} - \text{ERca}}{\text{ERexp}}\right) \times 100$$

In this relationship, n represents the number of experiments performed, while ERexp and ERcal correspond to the experimental and model-predicted removal efficiencies, respectively. Furthermore, a model is considered adequate if the coefficient of variation is less than 5% (Ncibi *et al*., 2008). The optimal activation conditions were determined using iso-response curves or response surface plots generated by Minitab 19 software. The optimal values of the selected variables were obtained using the desirability function available in Minitab, along with an analysis of the response surface contours.

Adsorption experiments: The removal efficiencies of ions by the prepared materials were determined through adsorption tests. These tests were carried out in batch mode by bringing 1 g of material into contact with 25 mL of a synthetic phosphate or nitrate solution at a concentration of 50 mg/L in a 100 mL Erlenmeyer flask. The pH was adjusted to 4.7 using a 1 N sulfuric acid solution or a 1 M sodium hydroxide solution, in accordance with previous studies (Dable *et al*., 2008). The mixture was then stirred for 2 hours using a magnetic stirrer at room temperature and a speed of 300 rpm. The solution was separated from the adsorbent by centrifugation at 400 rpm for 12 minutes. The resulting supernatant was analyzed by UV–Visible spectrophotometry at wavelengths of 880 nm for phosphate ions and 415 nm for nitrate ions to determine the residual concentration. The removal efficiency is given by the following equation (Eqn. 3):

$$E_R = \frac{(C_0 - C_e)}{C_0} \times 100$$

E_R: ion removal efficiency (%)
C_0: initial ion concentration (mg/L)
C_e: residual ion concentration (mg/L)

RESULTS AND DISCUSSION

Results of the Plackett–Burman screening design for phosphate ions: The results of the Plackett–Burman matrix are presented in Table 2 below. The adsorption test results for phosphate ions, under various experimental conditions, are evaluated based on the removal efficiency (ER%).The ER response is expressed as the mean of two replicates ± standard deviation. Analysis of Table 2 shows that the responses range from 39.80 ± 0.70% (test 2) to 57.15 ± 0.85% (test 1).

Table 2. Plackett–Burman experimental design

Tests	X1	X2	X3	X4	X5	X6	X7	ER(%)
1	CHE	A	4H	30	10	H_2SO_4	0,5N	57,15±0,85
2	CHE	B	2H	60	10	H_2SO_4	0,1N	39,80±0,70
3	CCA	B	4H	30	20	H_2SO_4	0,1N	49,95±0,65
4	CHE	A	4H	60	10	NaOH	0,1N	44,00±1,2
5	CHE	B	2H	60	20	H_2SO_4	0,5N	46,20±0,6
6	CHE	B	4H	30	20	NaOH	0,1N	48,00±1,15
7	CCA	B	4H	60	10	NaOH	0,5N	53,55±0,45
8	CCA	A	4H	60	20	H_2SO_4	0,5N	57,10±0,80
9	CCA	A	2H	60	20	NaOH	0,1N	49,00±0,30
10	CHE	A	2H	30	20	NaOH	0,5N	50,70±0,60
11	CCA	B	2H	30	10	NaOH	0,5N	52,70±1,30
12	CCA	A	2H	30	10	H_2SO_4	0,1N	51,05±1,65

The average values indicate a higher removal efficiency for cocoa (44.85% ± 0.58) compared to rubber (42.79% ± 0.64). This trend is observed in several individual tests, particularly tests 1 and 8 (cocoa activated with H_2SO_4 at 0.5 N), which show among the highest removal efficiencies (~57%). Activation with NaOH resulted in a slightly higher average removal efficiency (44.81%) compared to H_2SO_4 (43.17%). The results also show that more concentrated solutions (0.5 N) provide a higher average removal efficiency (44.98%) than those at 0.1 N (42.99%). The statistical data obtained from the screening of factors for biosorbent preparation from cocoa and rubber shells are presented in Table 3 below.According to the results presented in the table above, five (5) factors out of the seven studied have a significant influence on the removal efficiency of phosphate ions, as their p-values are lower than 0.05. These factors, in decreasing order of importance, are washing temperature (X4), activation time (X3), normality (X7), biomass type (X1), and particle size (X2). The type of activating agent, biomass type, and particle size are qualitative factors and were therefore fixed for the subsequent

analysis. The positive and negative coefficients indicate, respectively, that NaOH, cocoa shells, and smaller particle sizes are more favorable for achieving higher phosphate removal efficiency.

The ER response is expressed as the mean of two replicates ± standard deviation. Analysis of Table 5 shows that the measured removal efficiency (ER) ranges from 40.27% to 57.93%, indicating

Table 3. Statistical analysis of coefficients

Factor	X1	X2	X3	X4	X5	X6	X7
Coefficient	-2,313	-1,587	1,671	1,673	0,246	0,254	2,946
P-value	0,000	0,001	0,000	0,001	0,528	0,515	0,000

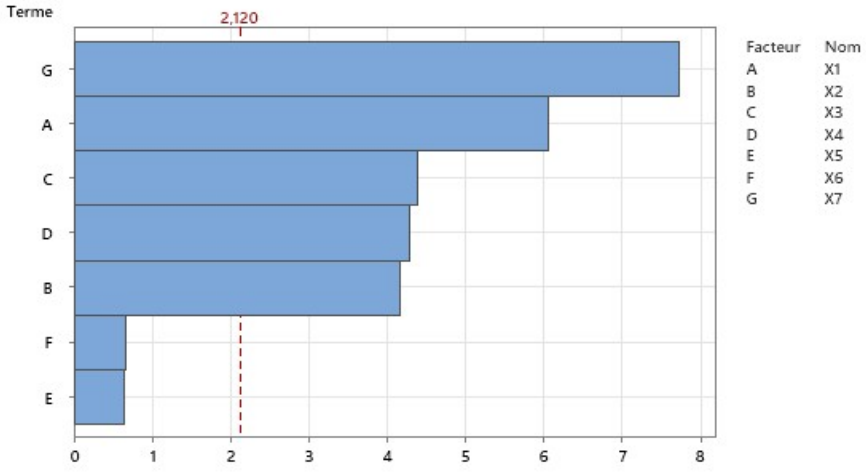

Figure 1. Pareto chart of standardized effects for phosphate ions

Table 4. Statistical indicators for model fitting and validation

Source	Value
Model	0,000
R^2	90,49%
R^2 ajusted	86,34%

Table 5. Plackett–Burman experimental design

Tests	X1	X2	X3	X4	X5	X6	X7	ER(%)
1	CHE	A	4H	30	10	H_2SO_4	0,5N	57,02±0,71
2	CHE	B	2H	60	10	H_2SO_4	0,1N	40,27±0,13
3	CCA	B	4H	30	20	H_2SO_4	0,1N	49,98±0,17
4	CHE	A	4H	60	10	NaOH	0,1N	50,28±1,12
5	CHE	B	2H	60	20	H_2SO_4	0,5N	49,96±0,81
6	CHE	B	4H	30	20	NaOH	0,1N	50,58±0,08
7	CCA	B	4H	60	10	NaOH	0,5N	49,87±0,17
8	CCA	A	4H	60	20	H_2SO_4	0,5N	57,93±0,14
9	CCA	A	2H	60	20	NaOH	0,1N	52,70±0,38
10	CHE	A	2H	30	20	NaOH	0,5N	56,76±0,27
11	CCA	B	2H	30	10	NaOH	0,5N	45,10±0,15
12	CCA	A	2H	30	10	H_2SO_4	0,1N	47,61±1,32

In the Pareto chart, the red vertical line represents the statistical significance threshold. The combined analysis of the Pareto chart and the coefficient table shows a perfect agreement in the statistical results. Indeed, the factors biomass type (X1), particle size (X2), activation time (X3), washing temperature (X4), and normality (X7) exhibit standardized effects greater than the critical value ($\alpha = 0.05$). In contrast, the factors ratio (X5) and type of activating agent (X6) do not exceed the significance threshold and show p-values well above 0.05, indicating a negligible influence on the removal efficiency. Table 4 presents the statistical indicators for model fitting and validation.The model significance value ($p = 0.000$) is lower than the threshold of 0.05. The coefficient of determination R^2 is equal to 90.49%, indicating that the model explains 90.49% of the total variability of the response. This result reflects an excellent goodness of fit. The adjusted coefficient of determination R^2_{adj} is 86.34%. This value, slightly lower than R^2, takes into account the number of parameters included in the model.

Results of the Plackett–Burman screening design for nitrate ions: The results of the Plackett–Burman matrix are presented in Table 5 below.

an overall efficiency from moderate to high depending on the operating conditions. This suggests that the process is capable of removing more than half of the targeted fraction in most experiments, reflecting a good performance of the applied treatment.Rubber shell (CHE) exhibits a higher average removal efficiency (50.81%) compared to cocoa shell (CCA), which shows an efficiency of 49%. The statistical data obtained from the screening of factors for biosorbent preparation from cocoa and rubber shells are presented in Table 6 below.According to the results presented in the table above, four (4) factors out of the seven studied have a significant influence on the removal efficiency of nitrate ions, as their p-values are lower than 0.05. These factors, in decreasing order of importance, are the mass ratio (X5), activation time (X3), normality (X7), and particle size (X2). Particle size is a qualitative factor and was therefore fixed for the subsequent analysis. The positive coefficients indicate that rubber shells, NaOH, and smaller particle sizes are more suitable for achieving higher nitrate removal efficiency.The analysis of the Pareto chart shows that the factors particle size (X2), ratio (X5), normality (X7), and activation time (X3) are statistically significant, with particle size (X2) appearing as the most influential parameter on the removal efficiency.

Table 6. Statistical analysis of coefficients

Factor	X1	X2	X3	X4	X5	X6	X7
Coefficient	0,140	-3,046	1,938	-0,503	2,314	0,209	2,102
P-value	0,579	0,00	0,00	0,058	0,01	0,410	0,00

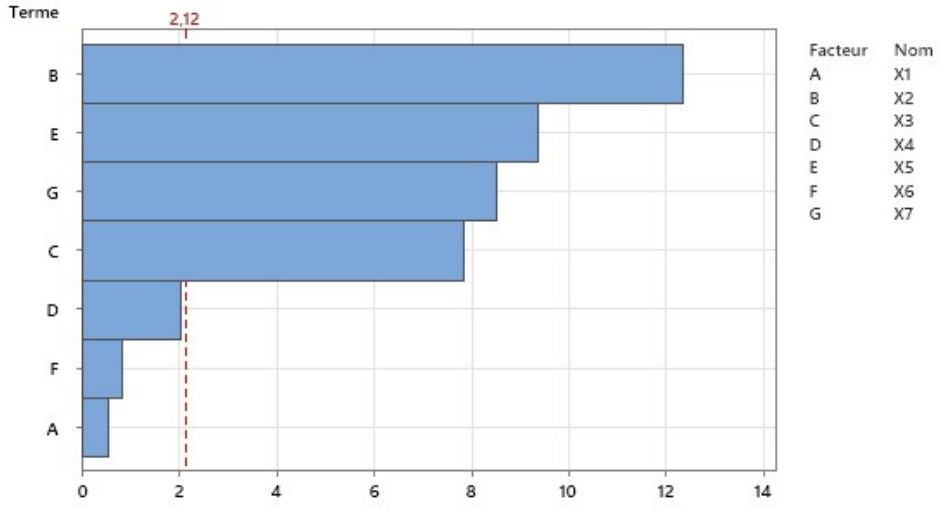

Figure 2: Pareto chart of the main effects of nitrate ions

Table 7. Model significance and statistical goodness-of-fit

Source	Valeur
Model	0,000
R^2	95,96%
R^2 ajusted	94,19%

In contrast, biomass type (X1) and type of activating agent (X6) exhibit effects below the critical threshold and can be considered non-significant in this model. Table 7 presents the significance of the model and the statistical goodness-of-fit.The coefficient of determination (R^2) reaches 95.96%, indicating that the model explains nearly 96% of the variability in the removal efficiency. Moreover, the adjusted R^2 (94.19%) is very close to R^2, with a difference of about 1.77%, which reflects the robustness of the model.

DISCUSSION

The results obtained highlight a marked selectivity of the studied biosorbents toward nutrient salts. Indeed, cocoa shell proves to be more effective for the removal of phosphate ions, whereas materials derived from rubber tree shells show a better affinity for the adsorption of nitrate ions. This difference in behavior can be explained by the physicochemical nature of the ions as well as the structural and chemical characteristics of the biosorbents (Nadagouda *et al*., 2024; Loganathan *et al*., 2014b).Furthermore, the small difference observed between the coefficient of determination (R^2) and the adjusted R^2 (approximately 4%) indicates a good fit of the model, suggesting the absence of overfitting and the relevance of the variables included. This observation is supported by the associated p-values, all below 0.05, confirming the statistical significance of the model and its ability to reliably explain the variability of the response. The high efficiency of cocoa shell toward phosphate removal can be attributed to its richness in oxygen-containing functional groups (hydroxyl, carboxyl, and phenolic groups), derived from its lignocellulosic structure. These functional groups promote the formation of coordination bonds, hydrogen bonding, and surface complexation phenomena with phosphate ions, leading to stable adsorption (Ahmed & Hayes, 2025; Crini, 2006b). In addition, the porous structure of the material increases the available specific surface area and facilitates the diffusion of ions toward active sites.Phosphate ions, characterized by a high negative charge and a polyatomic structure, preferentially interact with surfaces rich in active sites capable of forming strong interactions.

Previous studies have shown that these ions can even diffuse into the internal structure of certain biosorbents, such as date palm fibers (Riahi *et al*., 2009). Thus, the adsorption mechanisms involve both physical and chemical phenomena, which are particularly effective for multivalent anions (Foo & Hameed, 2010b; Loganathan *et al*., 2014b). This performance is especially important since phosphates are a major limiting factor in the eutrophication of continental aquatic environments. Their targeted removal therefore represents a key environmental challenge to limit algal proliferation and the degradation of water quality (Paerl *et al*., 2024).Conversely, nitrate ions, characterized by a single charge and high mobility in aqueous solution, are generally more difficult to adsorb. Their removal requires surfaces with suitable electrostatic affinity (Aini *et al*., 2024; Bhatnagar & Sillanpää, 2011b). Biosorbents derived from rubber tree shells meet these requirements due to their more homogeneous surface and the presence of active sites that promote weak but effective electrostatic interactions for this type of ion (He *et al*., 2023; Li *et al*., 2025). In addition, the surface pH of these materials can favor the protonation of functional groups, thereby enhancing the attraction of nitrate ions (Li *et al*.,2025). In this case, adsorption is mainly governed by ion exchange and physical adsorption mechanisms, which are better suited to monovalent and highly soluble anions such as nitrates (Bhatnagar & Sillanpää, 2011b).The analysis of experimental factors also shows that certain parameters, notably normality (X7), biomass type (X1), activation time (X3), and temperature (X4), have a significant influence on the response, unlike particle size and contact time, whose effects remain limited. This suggests the absence of notable diffusion resistance under the studied operating conditions, making the process effective even with fine particles and short treatment durations. Beyond the mechanistic aspect, nitrate removal also represents a major health concern due to the risks associated with their presence in drinking water, particularly methemoglobinemia (Aini *et al*., 2024;; WHO, 2017). Thus, the observed selectivity highlights the importance of adapting the choice of biosorbent to the targeted pollutant. Unlike conventional approaches aiming at a universal adsorbent, this study demonstrates that a strategy based on the complementarity of biosorbents makes it possible to optimize nutrient removal (Nadagouda *et al*., 2024).

Consequently, the combined use of biosorbents derived from cocoa shells for phosphates and rubber tree shells for nitrates appears to be a particularly promising integrated approach for the treatment of nutrient-rich waters. Such a strategy would enable the simultaneous reduction of phosphorus and nitrogen inputs, two key elements responsible for eutrophication.

CONCLUSION

This study evaluated the removal of nitrates and phosphates by biosorption using biosorbents derived from agricultural residues, namely cocoa shells and rubber tree shells, within the framework of sustainable water treatment and eutrophication control.The results highlighted a marked selectivity of the biosorbents. Indeed, cocoa shell proved more effective for phosphate removal, while rubber tree shell showed better performance for nitrate adsorption. This differentiation is attributed to the physicochemical properties of the ions as well as the surface characteristics of the biosorbents.These findings emphasize the value of a targeted and complementary approach, combining both biosorbents to achieve simultaneous reduction of nitrogen and phosphorus inputs. Finally, further optimization is envisaged to improve the efficiency of the biosorbents, along with morphological and structural characterizations to better understand their affinity toward different types of ions.

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