



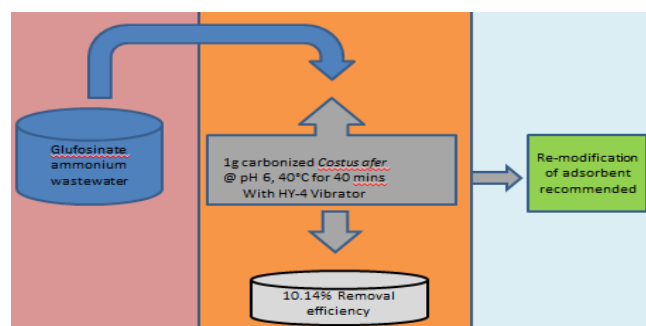
Adsorptive removal of glufosinate ammonium in wastewater using carbonized ginger lily (*Costus afer*)

Emmanuel Amuntse Yerima^{a,*}, Ntefreh Utomobong Percy^a, Edmund Luke Obasi^a and David Abutu^b

^a Department of Chemistry, Federal University Wukari, PMB 1020, Taraba State, Nigeria; ^b Department of Chemical Engineering, Federal University Wukari, PMB 1020, Taraba State, Nigeria

ABSTRACT

The alarming impact of the agrochemical, glufosinate ammonium, on aquatic ecosystems underscores the urgent need for effective remediation strategies. This study adopts the response surface methodology to find the optimum adsorptive removal of glufosinate ammonium in wastewater using carbonized *Costus afer* as adsorbent. Characterization of the adsorbent through FTIR revealed the presence of functional groups such as -OH, -C≡C-, -C=C- and C-N after carbonization which could aid its surface interaction with the agrochemical. SEM and TEM revealed the presence of minimal pore spaces, which easily get saturated during the adsorption process. EDX showed the introduction of Fe and Cu on the adsorbent after adsorption. The optimal removal efficiency of glufosinate ammonium by carbonized *Costus afer* was 10.14% which was quite low within the optimum condition (pH = 6, Temperature = 40 °C, Adsorbent dose = 1.0 g, Time = 60 min and rate of 0.01 g/L/min. Traceable to the fact that the adsorbent was not modified in any way. Further studies are recommended on the modification of carbonized *Costus afer* for better performance in glufosinate ammonium removal.



HIGHLIGHTS

- Glufosinate ammonium is a potent water contaminant.
- The urgent need for remediation strategies.
- Carbonized *Costus afer* can reduce glufosinate ammonium in wastewater.
- The material can be modified for greater adsorptive removal efficiency.

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1. Introduction

The invention and introduction of diverse agro chemicals have contributed immensely in the improvement of agricultural production thereby resulting in better soil fertility, more stable yield, high food quality and better resistance to pest and diseases among others as beneficial side-effect (Yerima et al., 2022). While on the other hand, the increased use of agro chemicals such as herbicides (weed killers) and insecticides in agricultural revolution could result in continues rise in concentration of chemical pollutants in fresh water reservoir due to the water run-off carrying them from the fields if not properly managed by either channeling the run-off and subjecting it to treatment (Chaudhry & Malik, 2017; Yerima et al., 2023).

Recently, an alarming impact of the agrochemical, glufosinate ammonium, on aquatic ecosystems underscores the urgent need for effective remediation strategies. It is a non-selective, broad-

spectrum herbicide widely used in agriculture and forestry for effective elimination of unwanted vegetation. The product is a fast-acting herbicide which acts by targeting glutamine synthetase, it disrupts both photorespiration and the light reaction during photosynthesis, leading to photo-reduction of molecular oxygen, which generates reactive oxygen species as well as accumulation of ammonia and eventual death of plant cell (Takano & Dayan, 2020).

The herbicide's inhibitory effect can also impact negatively on various bacterial populations involved in wastewater treatment. These include bacteria responsible for breaking down organic matter, nitrifying bacteria involved in ammonia removal and the denitrifying bacteria crucial for nitrogen removal (Vallejo et al., 2018).

Glufosinate ammonium is known to have a moderate persistence depending on the prevailing environmental condition but high solubility in water hence it is readily leach into surface and groundwater, depending on the local precipitation rates and soil

* CONTACT: Yerima, E. A; yerimaemmanuel@yahoo.com; Department of Chemistry, Federal University Wukari, PMB 1020, Taraba State, Nigeria
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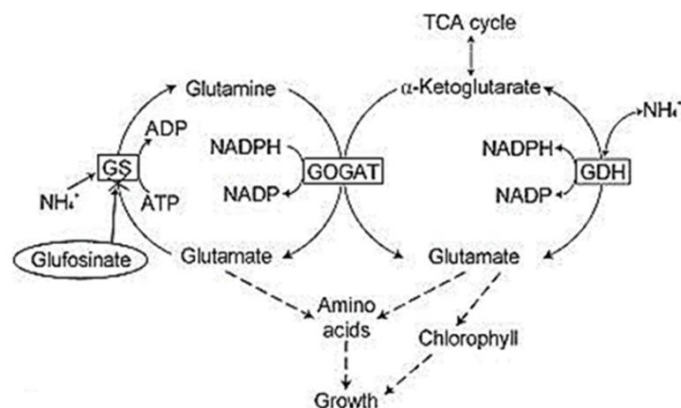


Figure 1. Mechanism of action of glufosinate ammonium adopted from Nolte et al. (2016).

permeability (Johnson, 2020). Even at low concentrations, glufosinate ammonium in wastewater can have a negative impact on aquatic organisms, leading to reduced survival, growth inhibition and reproductive abnormalities in various aquatic species, from moderate to high acute toxicity, leading to a decline in population and alteration of natural balance within the aquatic ecosystem (US EPA, 2018).

To manage the adverse effect of glufosinate ammonium, like other pesticides in waste water system, microbial degradation mechanisms does plays a crucial role in reducing the concentration of herbicides. However, for glufosinate ammonium, biotic degradation by microorganisms suffers a set back as it undergoes microbial metabolism to form several metabolites, some of which possess bactericidal properties capable of destroying microbial communities (Takano & Dayan, 2020).

Hence, the urgent need for other effective remediation strategies. For instance contaminants in wastewater systems have been managed by phytoremediation, photo-catalytic degradation, and adsorption with 80% to 90% removal efficiency. However, this is only possible at optimized conditions of factors such as pH, temperature, adsorbent dosage and presence of surface charged species and pores for sorption devoid of impurities (Omokheyeke & Vincent, 2021; Yerima et al., 2024). Fortunately, out of the diverse toolbox of technologies to combat this pollutant, membrane filtration techniques like reverse osmosis have been reported to be quite effective in removing glufosinate by approximately 76% at maximum, except for cost and energy considerations that needs to be addressed (Hinoon et al., 2023).

Phytoremediation takes a longer time to be implemented while most often, glufosinate ammonium degradation process may produce toxic metabolites as byproducts like 3-(hydroxymethyl phosphinyl) propionic acid (Masiol et al., 2018) which can be more harmful than the original compound; posing risk to both aquatic life and terrestrial organisms that depends on glufosinate ammonium contaminated water if not adequately removed (Tuzimski et al., 2016; Miller and Jones, 2021). This challenge necessitated a search into adsorption studies, which to the best of our knowledge, based on a literature search, only a few studies have reported on the adsorptive removal efficiency of glufosinate ammonium from aqueous solution by employing low-cost adsorbents. This necessitated a search into biochar from *Costus afer*, a rice weed and a tropical herbaceous, rhizomatous, monocotyledonous, relatively tall, unbranched plant belonging to the family Costaceae is grossly understudied. The plant is commonly known as ginger lily, spiral ginger, or bush cane and is widely distributed in nature

around shady forests of tropical West Africa (Nzambi et al., 2022; King et al., 2024). The plant extract is reportedly rich in antioxidant and inhibits microbial growth (Uchegbu et al., 2016); rich in phenolic compounds and inhibits the corrosion of mild steel (Adam et al., 2023). Traditionally, *Costus afer* has been used as medicine to treat diabetes, arthritis, stomach aches, and skin diseases (Boison et al., 2019). And has been reportedly rich in phytochemicals such as saponins, tannins, alkaloids, steroids, cardioglycosides, flavonoids, and terpenoids, making it a potential adsorbent (Nzambi et al., 2022). The utilization of *Costus afer* as a precursor for the production of activated carbon remains scarce in the literature. However, its activated carbon has been reportedly applied in the removal of methylene blue dye from aqueous solution (King et al., 2024).

Thus far, this study aims to adopt a low-cost strategy of adsorptive removal of glufosinate ammonium using *Costus afer* biochar. The optimization of key factors influencing glufosinate ammonium adsorption such as the amount of adsorbent, solution pH, temperature, and contact duration was conducted using the Box-Behnken design in the context of response surface methodology. The strategy that will simply adsorbed glufosinate ammonium without the need for degradation of the product into more harmful byproducts by simply employing a biochar from *Costus afer*.

2. Materials and Methods

2.1. Materials

Cycling vibrator (Model HY-4), weighing balance (Model PGW 453i), water bath (Model Clifton 98006), pH meter (Model Starter 2100), furnace (Model SX-5-12), beakers, conical flask, whatmann filter paper (90 mm diameter; pore size 11 µm), HCl (0.1M), NaOH (0.1M) for pH adjustment, plastic funnels, measuring cylinder were employed.

A non-selective herbicide with the name Dragon Super a product of Wacot Nigeria limited (Batch Number: 20230825, NAFDAC Reg Number: A10-100285) with active ingredient glufosinate ammonium (200 g/L) was used as adsorbate. The product was purchased from an agrochemical firm (NICOL) at Wukari, Nigeria.

2.2. Collection of *Costus afer*

Growing *Costus afer* plants were obtained from Ikot Inyang village, Oruk Anam Local Government Area of Akwa Ibom State where the plant is widely distributed. Transported to Wukari, Taraba State, identified in the Department of Biological Sciences at Federal University Wukari, replanted, watered, and monitored for 12 weeks, before it was harvested. The plant sample was washed by means of running water, rinsed with deionized water, separated into leaves, stem, root and air dried at room temperature for two weeks.

2.3. Thermal activation of *Costus afer*

The method used was adopted from Reza et al. (2020). Pre-weighed *Costus afer* sample (stem) cut into smaller pieces with a particle size of 5–15 mm by means of a small axe. The pieces were transferred into a pre-cleaned crucible, carbonized using a Muffle furnace at 300 °C for 1 hour, and cooled then transferred to a desiccator to stabilize the product for approximately 20 min. After which the crucible was removed, weighed and the carbonized material transferred into two different containers, one labelled carbonized *Costus afer* sample before adsorption while the other

was employed for the adsorptive studies and afterwards labelled carbonized *Costus afer* sample after adsorption.

2.4. Adsorptive experimental design

The experiment was designed by means of Design-Expert (version 13), adopting response surface, design types: box-behnken and quadratic design model due to its ability to generate higher order response surfaces using fewer required runs than a normal factorial technique.

The independent variables for the glufosinate ammonium removal process considered include: pH, temperature (°C), contact time (min) and dosage of *Costus afer* biochar (g) based on three levels coded as +1, 0, -1. And the coded values of the independent variables X_i were calculated by utilizing equation I (Hasanzadeh et al., 2020; Najafi and Rahimi, 2022).

$$X_i = \frac{\varepsilon_i - [\varepsilon_{Hi} + \varepsilon_{Li}]/2}{[\varepsilon_{Hi} - \varepsilon_{Li}]/2} \quad (I)$$

where X_i is the coded values of the independent variables. The high and low levels of independent criteria are ε_{Hi} and ε_{Li} , respectively. And Y is the dependent criterion equation of the quadratic model shown in equation II.

$$Y = \beta_0 + \sum_{i=1}^4 \beta_i X_i + \sum_{i=1}^4 \beta_{ii} X_i^2 + \sum_{i=1}^3 \sum_{j=2}^4 \beta_{ij} X_i X_j \quad (II)$$

where the independent variables are denoted as X_i and X_j , and the regression coefficients correspond to the constant term, linear term, quadratic term, and interaction term, represented by β_0 , β_i , β_{ii} , and β_{ij} , respectively.

The level of Central Composite Design experiments considering the four effective independent parameters for the removal of glufosinate ammonium *Costus afer* biochar include: pH (3, 6 and 9); temperature (20, 40 and 60 °C); adsorbent dose (0.5, 1.0 and 1.50 g) and adsorption time (30, 60 and 90 min). A total of 25 experimental runs were randomly selected and recommended by the design expert, as shown in Table 1.

2.5. Batch adsorptive removal of glufosinate ammonium from wastewater

Stock of glufosinate ammonium (35 mL) with a concentration of 200 g/L and the trade name 'Dragon Super' was measured into a 1000 mL capacity volumetric flask and diluted with deionized water to the mark, giving an initial concentration of 7 g/L. To 50 mL of the 7 g/L glufosinate ammonium solution in a 100 mL conical flask, 1.0 g of the carbonized *Costus afer* (adsorbent) was added and mixed using a stirring rod. The experimental pH of the mixture was adjusted to 3 using HCl (0.1 M) and NaOH (0.1 M) with the aid of a pH meter before it was taken to a water bath set at 40 °C, allowing the mixture to assume the same temperature. Subsequently, the removal of glufosinate ammonium by carbonized *Costus afer* was performed using a cycling vibrator set at 800 rpm over a contact time of 30 minutes before filtering, based on the experimental design displayed in Table 1.

The same procedure was replicated for the other 24 experiments based on the conditions displayed in Table 1 and the sample containers properly labelled following the design. The residual concentration of glufosinate ammonium in the 25 filtrate were determine using ultraviolet visible spectrophotometer while the adsorbent residue after adsorption was characterized and

compared with the carbonized *Costus afer* adsorbent before the adsorption process.

2.6. Characterization of carbonized *Costus afer* before and after adsorption

The functional groups in *Costus afer* biochar (adsorbent) before and after adsorption were determined using the Pelkin Elmer 3000 MX FTIR Nicolet after mixing few samples with KBr. The spectra were recorded with 32 scans at a resolution of 4 cm⁻¹ in the range of 4000-400 cm⁻¹. While the IR spectra were analysed using the spectroscopic software Win-IR Pro Version 3.0 with a peak sensitivity 2 cm⁻¹ (Giechaskiel et al., 2021). The TEM spectrometer and SEM (JSM 6701F (JOEL) were used to understand the surface morphology where the sample was placed onto the specimen stub and coated with platinum evaporative coating under high vacuum. It was operated at 15 kV with 15 mm working distance (Minuti et al., 2023). The energy dispersive x-ray analysis (EDX) spectrometer was employed to determine the elemental composition of the adsorbent (Khan et al., 2021).

2.7. Determination of glufosinate ammonium residual concentration

The calibration curve for the concentration versus absorbance of glufosinate ammonium was obtained using standard solutions of glufosinate ammonium at concentrations of 0, 0.5, 1, 1.5, and 2.0 g/L. The corresponding absorbance values were determined using a UV-visible Spectrophotometer (UV-6300PC) set at 291 nm, with the equipment blank using distilled water. The UV-visible Spectrophotometer (UV-6300PC) was also used to determine the absorbance and the corresponding residual concentrations of filtrates from the 25 experimental runs. Subsequently, the removal

Table 1. Experimental design and conditions

Run	Dosage (g)	Temperature (°C)	pH	Contact time (min)
1	0.5	20	6	60
2		40	3	60
3		40	6	30
4		40	9	60
5		60	6	60
6	1.0	20	3	60
7		20	3	90
8		20	6	30
9		20	9	60
10		40	3	30
11		40	3	90
12		40	6	60
13		40	9	30
14		40	9	90
15		60	3	30
16		60	3	60
17		60	6	90
18		60	9	60
19	1.5	20	6	60
20		40	3	60
21		40	6	30
22		40	9	60
23		40	6	60
24		40	6	90
25		60	6	60

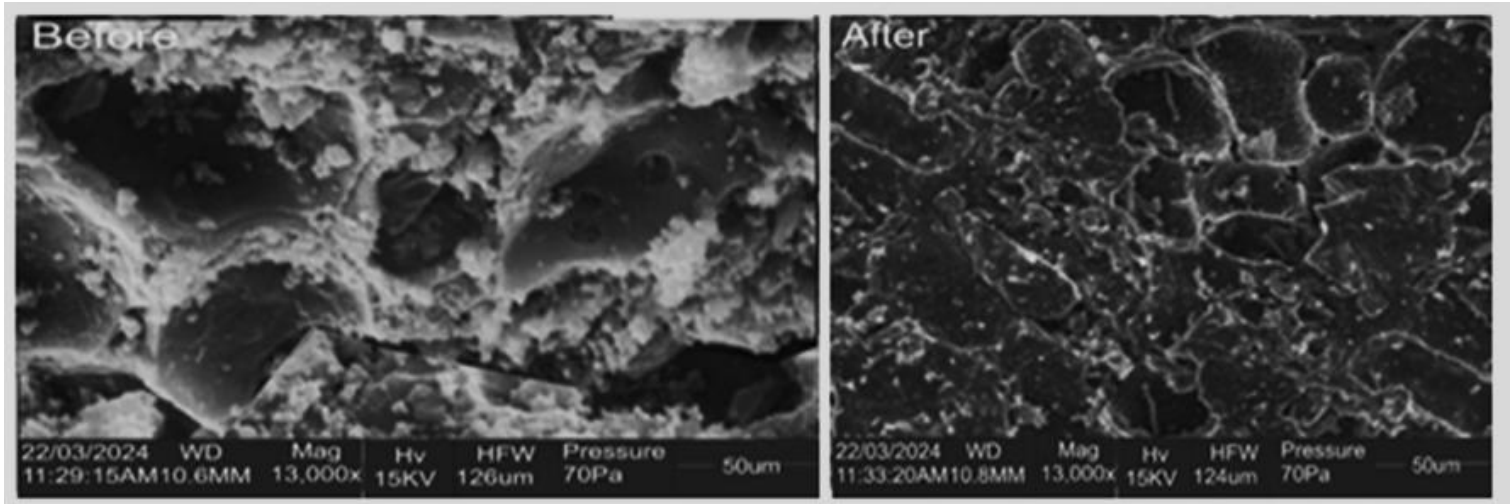


Figure 2. Scanning electron micrograms of carbonized *Costus afer* at 50 μm magnification before and after adsorption

efficiencies of each experimental run were calculated, as well as the adsorption rate with respect to time.

2.8. Data analysis

The adsorption capacity (Q_e) and adsorptive removal efficiency (R_e) were calculated using the concentration difference between the initial concentration and the equilibrium concentration displayed in equations III and IV. Where C_o is the initial concentration of ammonium, C_e is the equilibrium concentration of ammonium, M is the mass of adsorbent (carbonized *Costus afer*), and V is the solution volume (Seliem et al., 2013). The rate of glufosinate ammonium removal was estimated by dividing the amount of glufosinate ammonium adsorbed by the respective contact time shown in equation V.

$$Q_e = \frac{C_o - C_e}{M} \times V \quad (\text{III})$$

$$R_e = \frac{C_o - C_e}{C_o} \times 100 \quad (\text{IV})$$

$$\text{Rate of adsorption} = \frac{C_o - C_e}{t} \quad (\text{V})$$

Results and Discussion

2.9. Characterization of *Costus afer* biochar (adsorbent)

The surface morphological evaluated by means of SEM of *Costus afer* biochar at 50 μm magnification displayed in Figure 2 showed pores with circular holes which are more visible before analysis than after analysis. The reduction in visibility of these pores shows that *Costus afer* provide reactive sites which easily get saturated during adsorption process.

The TEM micrograph of the carbonized *Costus afer* shown in Figure 3 at 100 nm reveals the presence of few measurable particles within the range of 1.32 nm to 8.32 nm which were spherical in shape and well dispersed while after adsorption the number of measurable particles increased from 7 to 12 within the range of 1.32 nm to 5.32 nm suggesting adsorption of particles to the surface of the adsorbent.

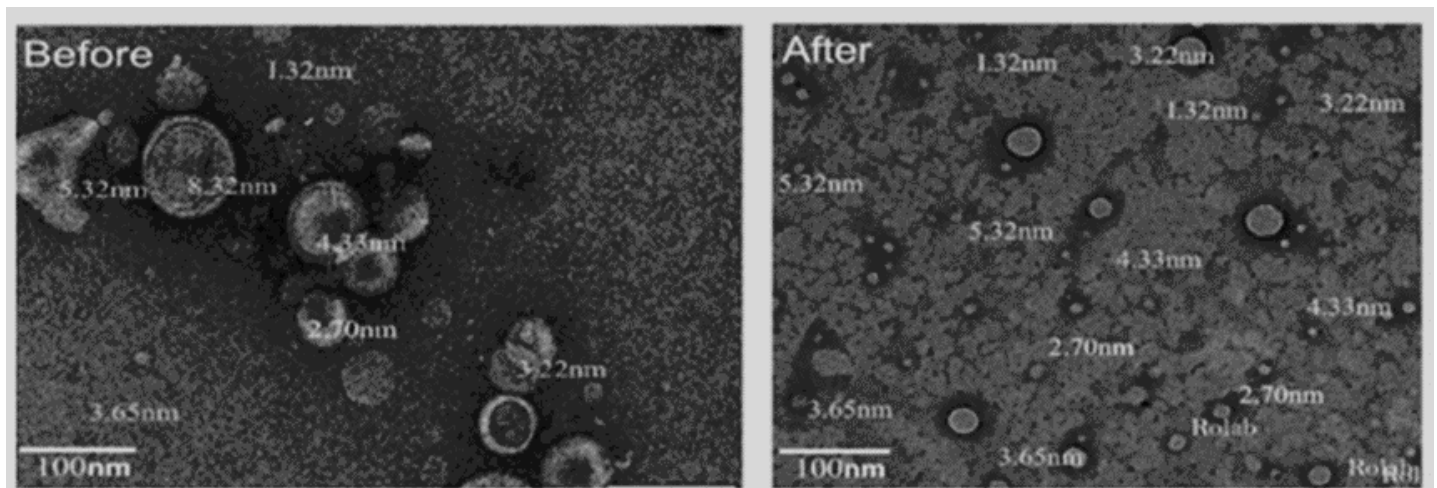


Figure 3. Transmission electron microgram of carbonized *Costus afer* before and after adsorption at 100 nm magnification

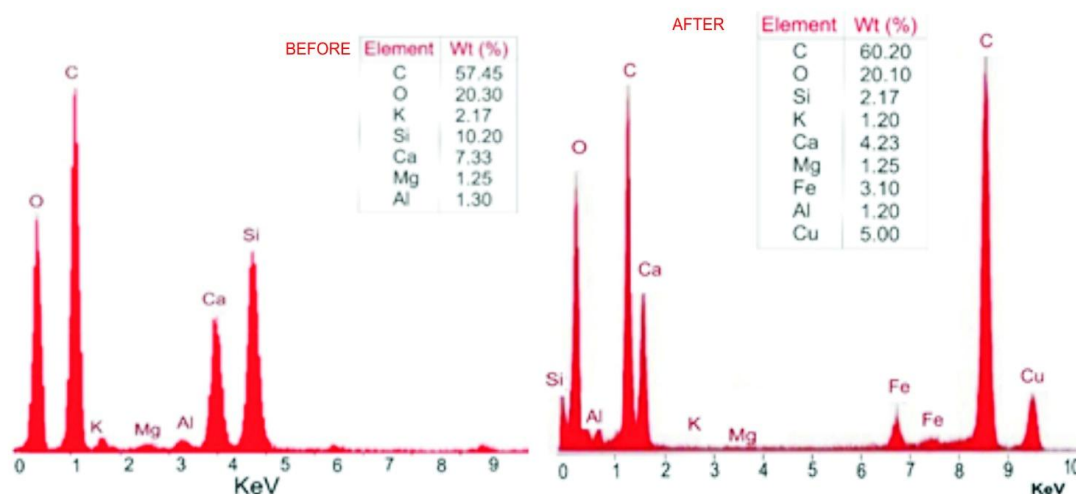


Figure 4. Energy Dispersive X-ray spectra of carbonized *Costus afer* before and after adsorption

The EDX was used to characterize the adsorbent before and after adsorption, and the outcome displayed in Figure 4. Before adsorption, the elements found in carbonized *Costus afer* were C, O, K, Si, Ca, Mg and Al in the various proportions of 57.45%, 20.30%, 2.17%, 10.20%, 7.33%, 1.25% and 1.30%. Carbon had the highest value of 57.45% while Magnesium had the lowest value of 1.25%.

However, after the adsorption process, the EDX spectra revealed the presence of additional elements: Fe and Cu were also found in the proportions of 3.10% and 5% on the adsorbent, an indication that the adsorbent may find utility in the adsorptive removal of Fe and Cu which are present in the adsorbate (herbicide) in trace amount. Since the utilization of *Costus afer* as a precursor for the production of activated carbon and its utilization in adsorption studies of Fe and Cu remains relatively scarce in literature (King et al., 2024). The studied adsorbent may be employed in further studies to ascertain the hypothesis.

FTIR spectrum presented in Figure 5 indicates the presence of C, H and O bonds interactions which may be single, double or triple in nature and all plays a role in influencing the level of

adsorption (Kibami et al., 2014). A careful observation of the FTIR spectrum before and after adsorption shows a general shift (decrease) in wave number of the functional groups after adsorption. While the functional groups suspected to be present were O-H, C-H, C-N, and -C=C-, a decrease was observed in their wave numbers. Specifically, the O-H group showed a decrease from 3445.00 to 3436.00 cm^{-1} , while the C-H group showed a decrease from 2927.76 to 2926.54 cm^{-1} . Additionally, a decrease was also observed in the -C=C- group, from 1631.73 to 1630.66 cm^{-1} . These shifts in wavenumber indicate a change in the vibrational frequency of a specific bond or functional group within the molecule. This change can be due to factors such as a change in bond strength, changes in the surrounding molecular environment. The various changes observed in the spectrum before and after analysis is an indication of effective surface interaction between the adsorbate and the adsorbent during the adsorption process (Kibami et al., 2014).

2.10. Adsorption capacity and removal efficiency

The residual concentration, amount adsorbed adsorption capacity and removal efficiency of glufosinate ammonium after the adsorption process of the 25 experimental runs is displayed in Table 2. The result obtained shows that the optimal removal efficiency was 10.14% with corresponding adsorption capacity of 19.95 g/L. This was achieved at pH of 6, temperature of 40°C, dosage of 1.0 g and contact time of 60 minutes. However, this was less than the 22% removal of glufosinate by means of open-circuit adsorption as well as far less than the 94.9% removal by means of electrosorption (Tongur and Ayranci, 2023).

The low adsorption capacity of the adsorption medium in this experiment is due to the fact that the adsorbent used is unmodified in any way (Li et al., 2022).

However, significant post-treatments of the carbonized *Costus afer* by either acidic or basic treatment can enhance both the textural and physico-chemical properties of the adsorbent by incorporation of surface functionalities and getting rid of surface interferences, thereby positioning the material for real-time application in higher scale (Petrovic et al., 2022).

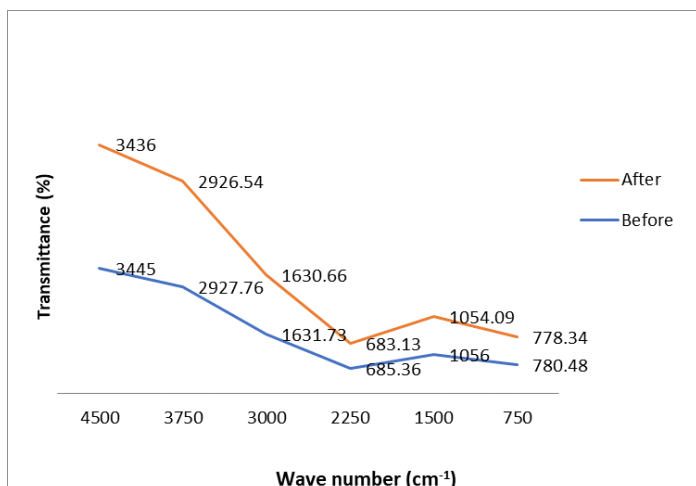


Figure 5. FTIR spectrum of *Costus afer* biochar before and after adsorption

Table 2. Glufosinate ammonium concentration (g/L) and its removal efficiency

Run	C ₀	C _e	Amount Adsorbed	Adsorption Rate	Adsorption capacity	R _e (%)
1	7.00	6.51	0.49	0.01	17.15	7
2	7.00	6.29	0.71	0.01	16.56	8.14
3	7.00	7.00	0.00	0.00	0	0
4	7.00	6.97	0.03	0.0005	1.05	0.42
5	7.00	6.98	0.02	0.0006	0.7	0.28
6	7.00	6.97	0.03	0.0005	2.1	0.64
7	7.00	7.00	0.00	0.00	0.0	0
8	7.00	7.00	0.00	0.00	0.0	0
9	7.00	6.97	0.03	0.001	1.05	0.42
10	7.00	6.97	0.03	0.0003	0.69	0.42
11	7.00	6.97	0.03	0.0005	0.69	0.42
12	7.00	6.97	0.03	0.0005	2.1	0.42
13	7.00	6.96	0.04	0.0006	1.4	0.57
14	7.00	6.97	0.03	0.0003	1.05	0.42
15	7.00	6.43	0.57	0.0095	19.95	10.14*
16	7.00	7.00	0.00	0.00	0	0
17	7.00	6.96	0.04	0.0004	1.4	0.57
18	7.00	6.97	0.03	0.0003	1.05	0.42
19	7.00	6.97	0.03	0.0003	2.1	0.42
20	7.00	6.98	0.02	0.0003	0.46	0.28
21	7.00	6.97	0.03	0.0005	2.1	0.42
22	7.00	6.97	0.03	0.001	2.1	0.42
23	7.00	6.97	0.03	0.0003	1.05	0.42
24	7.00	6.98	0.02	0.0003	0.7	0.28
25	7.00	6.97	0.03	0.0005	2.1	0.42

* = Optimum removal efficiency, C₀ = Initial concentration, C_e = Residual concentration, R_e = Removal efficiency

2.11.Effect of temperature, adsorbent dosage, pH, and contact time on the removal of glufosinate ammonium

The effect of temperature, adsorbent dosage, pH and contact time on the removal efficiency of glufosinate ammonium are shown in Figures 6 and 7 revealed that as the temperature increases from 20 °C to 40 °C, the removal efficiency also increases from 2 to 8.14% but as the temperature increases further from 40 °C to 60

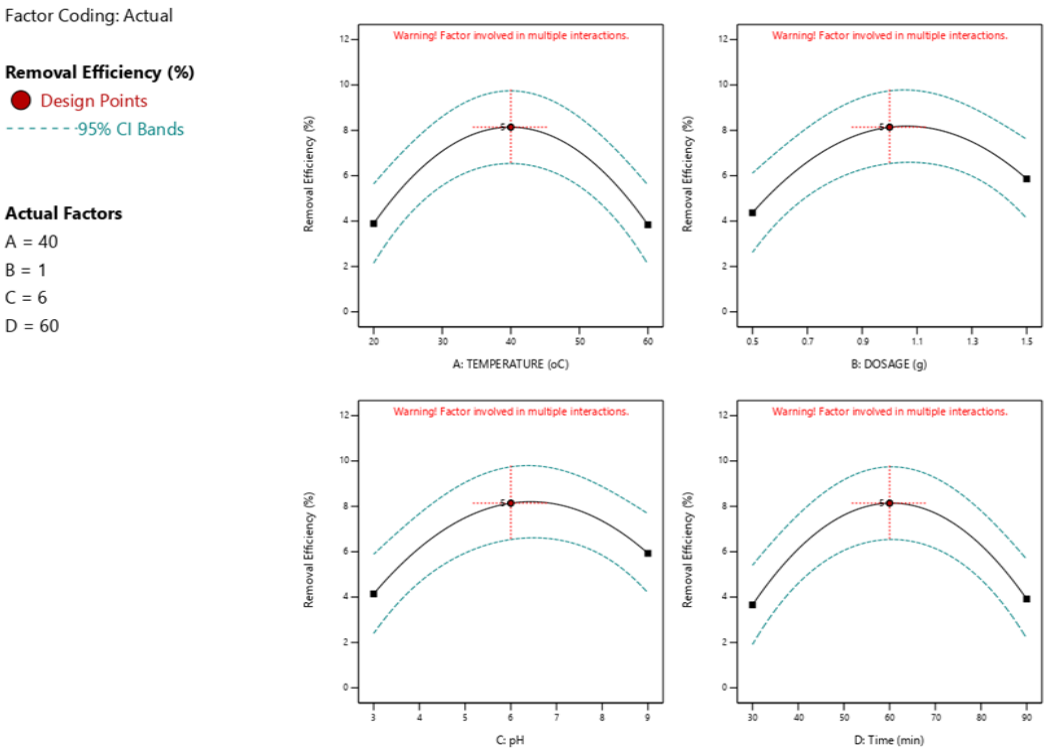


Figure 6. Effect of temperature, adsorbent dosage, pH, contact time on removal of glufosinate ammonium

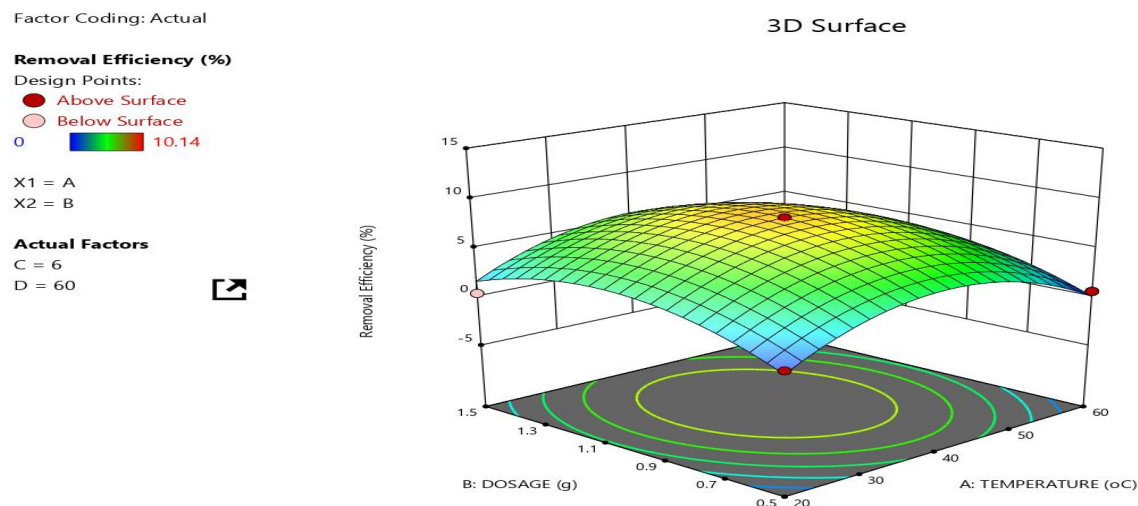


Figure 7. Effect of dosage and temperature on the removal efficiency of glufosinate ammonium

°C there was an observed decrease in the removal of glufosinate ammonium indicating 40 °C as the optimum temperature for the adsorption process. The same trend was observed as the adsorbent dosage increased from 0.5 g to 1.0 g. The removal efficiency of glufosinate ammonium also increases from 2.0% to 8.14% but as the dosage increased beyond 1.0 g, there was an observed decrease in removal efficiency. Likewise, the optimum pH of 6 and contact time of 60 min correspond with the maximum 10.14% removal efficiency of glufosinate ammonium.

The optimum condition for the removal of glufosinate ammonium by carbonized *Costus afer* was achieved at a temperature of 40 °C, adsorbent dosage of 1.0 g, pH of 6 and contact time of 60 minutes as beyond these set conditions remarkable decrease was observed in the removal efficiency.

Recently, Design Expert, as a collection of statistical and mathematical techniques, has been efficiently incorporated in a number of adsorption studies to determine and suggest the number of experimental runs to be carried out, as well as in the optimization of critical parameters with great precision (Madondo & Chetty, 2022; Yerima et al., 2023). However, out of the 25 experimental runs suggested by the design expert, no room to set all the critical parameters constant while varying one of interest; likewise, the number of data points for fitting models was insufficient.

3. Conclusion

Carbonized *Costus afer* is rich in functional groups such as -OH, C=C and C-N with minimal pore spaces which easily gets saturated during adsorption process. Through optimization of adsorption parameters using box-behnken and quadratic design model within Response Surface Methodology; The carbonized *Costus afer* is capable of removing glufosinate ammonium from wastewater systems with the adsorption capacity of 19.95 g/L and corresponding removal efficiency of 10.14% at the set optimum condition (pH = 6, Temperature = 40°C, Adsorbent dose = 1.0 g, Time = 60 min and rate of 0.01 min⁻¹. Since EDX analysis also showed the introduction of new elements like Fe and Cu on the adsorbent after adsorption, further studies is therefore recommended on, metal analysis of the product. While carbonized *Costus afer* should be modify by either acid activation or doping

with metal to improve adsorption efficiency of glufosinate ammonium.

CRedit authorship contribution statement

EAY: Resources, methodology, data collection and entry, data analysis, writing draft. **NUP:** Methodology, field work, data analysis, writing draft, reviewing and editing. **ELO:** Methodology and sample analysis. **DD** Methodology, data analysis, writing draft, reviewing and editing, supervision, visualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

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