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Polymer Composite-Supported Jackfruit Peel Adsorbent for Enhanced Copper (II) Removal from Wastewater: Batch Equilibrium and Isotherm Analysis

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Abstract

The increasing concern of disposal of copper-containing effluents into the environment has led to heightened interest in designing cost-effective, eco-friendly and scalable adsorbent systems. Traditional metals and synthetically engineered sorbents prove to be effective; however, these systems are energy intensive. Agricultural residues entrapped in polymer composite systems constitute an economically feasible solution to this environmental challenge. This work reports on the design, characterization and performance evaluation of an economical adsorbent prepared from jackfruit (Artocarpus heterophyllus) peel and its application in removal of copper ions from synthetic wastewater samples. The adsorbent was prepared after thorough washing, drying and calcination of jack fruit peels at temperatures ranging from 400°C to 450°C resulting in three particle sizes of 0.106 mm, 0.212 mm and 0.300 mm. The use of naturally available biopolymers, primarily pectin, cellulose and hemicellulose present in jack fruit peels as a base for polymer composite adsorption has been compared in this paper to the existing technology used for designing polymer composite adsorbents based on chitosan, functionalized poly(gamma-glutamic acid) and cellulose-based composites in recent studies. Batch adsorption experiments have been performed with different particle sizes, adsorbent doses (1-3g) and varying copper ion concentrations (100-300 mg/L) at pH=4.2 and 1000 rpm. Adsorption equilibrium concentrations of copper were quantified using UV-visible spectroscopy. Both the Langmuir and Freundlich adsorption equilibrium models were used to fit the experimental data, followed by regression analysis to obtain parameters for the respective adsorption isotherms. The maximum percentage of copper removed experimentally is 84.30%, while the maximum adsorption equilibrium capacity is 30.98 mg/g using a dosage of 1 g of adsorbent. Experimental data was successfully modeled using both isotherms, which indicated a heterogenous multilayer surface as well as monolayer chemisorption capacity, characteristics of natural biopolymer composite adsorbents. Such results confirm the viability of utilizing calcined jackfruit peel, which can be considered a naturally derived biopolymer composite material, as a potential component of engineered polymer matrix composites for metal ion removal from industrial wastewater effluents.

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Keywords: Adsorption, copper removal, jackfruit peel, polymer composite adsorbent, Langmuir isotherm, Freundlich isotherm, biopolymer, industrial waste water treatment, low-cost adsorbent, UV-spectroscopy

INTRODUCTION

The pollution of water sources by heavy metal ions continues to be a significant threat to the environment around the globe. Among the common pollutants emitted during plating operations, mining, semiconductor production, and metal finishing processes, copper emerges as a pollutant

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that garners significant attention due to regulatory concerns. Despite being an important constituent of biological organisms, the excess presence of copper over certain permissible limits can cause health hazards. According to WHO, the recommended guideline value of copper in drinking water is 2 mg/L, while the maximum contaminant level (MCL) prescribed by U.S. EPA is 0.25 mg/L. Over this limit, it causes acute health problems such as gastrointestinal disorders, liver and kidney toxicity, and neurological complications [1, 3, 6]. The persistence of Cu(II) ion in water bodies and its bioaccumulation through the food chain make it a pollutant of significant concern that needs removal from the effluents of these industries.

Existing methods of water purification include chemical precipitation, ion exchange, reverse osmosis, electrodialysis, and membrane technology and can effectively decrease the content of heavy metals to safe levels. However, all of them are associated with significant disadvantages due to the relatively high capital and operational costs, secondary sludge formation, and an increasing demand for energy during the processing of dilute metal solutions [4, 8]. As an alternative approach or a supplementary technique, adsorption technology deserves extensive consideration, especially if the adsorbent can be obtained from locally available agricultural by-products, which are readily available, cheap, and underutilized [2, 7].

The shift that radically changed the field of research on adsorption processes over the last decade was the increased awareness that agricultural biomass is more than just a carbonaceous skeleton with numerous pores; instead, it is a polymer composite structure that consists of cellulose, hemicellulose, pectin, and lignin. The carboxylate, hydroxyl, and amine groups contained in the mentioned biopolymers act as binding sites that interact with heavy metal ions [5, 16]. It led to the rapid development of polymer composite adsorbents, which were created based on natural or synthetic polymers, modified using fillers, cross-linking agents, or functional nanomaterials [20, 21].

Chitosan, obtained through deacetylation of chitin, has become one of the most extensively researched biopolymeric adsorbents for Cu(II) removal in aqueous medium. In their review, Mallik et al. [16] showed that chitosan functionalisation via crosslinking and grafting polymerisation enhances adsorption properties substantially, with Langmuir monolayer adsorption capacities surpassing 135 mg/g for thiourea crosslinked chitosan–silica composites. Suyambulingam et al. [17] demonstrated that adsorption capacities of chitosan nanocomposites containing Fe3O4, TiO2, or graphene oxide are between 204–378 mg/g for lead and copper adsorption, where the process follows pseudo-second-order kinetics and Langmuir isotherm. Likewise, in the work by Yilmaz et al. [18], the authors revealed that poly(gamma-glutamic acid) hydrogels spontaneously and exothermically adsorb Cu2+, based on pseudo-first-order kinetics and Langmuir isotherm, qualifying them as recyclable polymer adsorbents for industrial effluents treatment.

Apart from chitosan systems, heterocyclic polymers and biopolymer composites have been garnering increasing interest in recent years among researchers engaged in heavy metals adsorption. In their review, Oladoye et al. [19] pointed out that nitrogen and sulphur heteroatoms in heterocyclic polymers serve as chelating agents towards divalent metal ions, allowing them to act as selective and efficient adsorbents even at low metal concentrations. On the other hand, Saravanan et al. [20] proved through extensive literature reviews that using polymer composite materials in combination with extraction and membrane technologies allows process intensification for wastewater treatment at competitive costs. The review paper published in the npj Materials Sustainability journal by Elgarahy et al. [21] added more weight to the results indicating the superiority of chitosan-, keratin-, and cellulose-based biopolymer composites as effective adsorbents with an additional potential for being environmentally friendly and applicable in circular economies.

The use of fruit peels as adsorbents represents another area where these research trends converge. Biopolymers forming the basis for fruit peels, which comprise high amounts of pectin (polysaccharide that has free carboxylates) and cellulose fibers, act as natural matrices used to form metal ion complexes

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and undergo ion exchange similar to functionalized synthetic polymers [16, 18]. This fact was illustrated in the work done by Yadav et al. [22] who revealed that raw and acid/base-treated fruit peels, namely those from jackfruit (Artocarpus heterophyllus), were highly efficient in removing Cu(II) and Zn(II) from aqueous solutions due to their ability to interact via carboxylate (–COOH) and hydroxyl (–OH) groups confirmed via infrared spectroscopy analysis. Cu(II) removal efficacies of biochar produced from jackfruit peel and seed were reported to be as high as 99.84% when the conditions were optimized [23]. In addition, a magnetite-biochar nanocomposite synthesized from jackfruit peel material confirmed the feasibility of designing functionally enhanced biopolymer composite adsorbents using this abundant agricultural resource [24].

Although this list of examples continues to grow, the development of agricultural peel-based adsorbents from the perspective of biopolymer composites still receives relatively little attention. This means that only a small number of researchers attempt to interpret agricultural peels as biopolymer composites, where the peel matrix serves as the polymer phase, while the carbonaceous chars resulting from calcination processes serve as the filler phase. Such an interpretation has practical implications since it provides an opportunity to deliberately design novel adsorbents through additional treatment, including cross-linking, grafting functional groups, or doping with chitosan or polyacrylamide [16, 18, 20].

This paper attempts to address this issue by examining the behavior of Cu(II) adsorbed onto calcinated jackfruit peel within the context of biopolymer composites. All experimental results, from the impact of particle sizes, doses, initial concentrations, and isotherm constants, are documented without reservation, and the discussions relate all these findings to the surface chemistry of the polymer and adsorption principles for biopolymer composite systems. The main purposes of this study include the following: (i) to determine the efficiency of adsorption using the calcined jackfruit peel adsorbent in different process conditions; (ii) to model the equilibrium data according to Langmuir and Freundlich models and determine the isotherm constants; and (iii) to explain the findings using polymer composite adsorption theory.

LITERATURE SURVEY ON POLYMER COMPOSITE ADSORBENTS FOR CU(II) REMOVAL

There has been a tremendous increase in research activities on the development of polymer composite adsorbents for the removal of heavy metals from aqueous media in the past decade. Based on a comprehensive review of the recent literature indexed by SCIE/SCOPUS, a broad range of polymer matrices such as biopolymers (chitosan, cellulose), as well as synthetic polymers (polyimides, polyacrylamide) were explored as potential Cu(II) sorbents, and both Langmuir and Freundlich adsorption isotherms were used to analyse their equilibrium adsorption characteristics [16–21].

Table 1 shows the highlights of the selected research articles. The compiled information serves two purposes in relation to the present study. Firstly, it highlights the variety of polymer matrices and composite structures investigated by other researchers. Secondly, it sets the standard for the adsorption capacity of the materials studied herein in light of its unique features as a natural biopolymer composite. In this regard, it is interesting to note that all man-made polymer composites mentioned in Table 1 followed the Langmuir and/or Freundlich adsorption model. It is worth mentioning that the experimental results obtained in this study were also fitted to the Langmuir and Freundlich adsorption models [16–25].

The findings presented in Table 1 reveal an underlying mechanism common to all polymers composite adsorbents, where the Langmuir equation describes monolayer chemisorption on active sites of functional groups, while Freundlich equation characterizes the more heterogeneous surface resulting from composite formation. Thus, the observed adherence to both types of isotherms, similar to the calcined jackfruit peel adsorbent, used in the current research, is a natural phenomenon resulting from the composite properties of the biopolymer adsorbent rather than an exceptional case.

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Table 1. Summary of recent polymer composite and biopolymeric adsorbent studies for Cu(II) removal.

Ref.	Adsorbent / Polymer System	Metal Ion	Key Finding	Isotherm Model
[16]	Thiourea/chitosan/SiO2 composite beads	Cu(II)	qmax = 135.1 mg/g; pH 3 optimal; pseudo-2nd order kinetics	Langmuir
[17]	Chitosan biopolymer + Fe3O4, TiO2, graphene oxide nanocomposites	Cu(II), Pb(II)	qmax 204-378 mg/g; recyclable; pseudo-2nd order kinetics	Langmuir / Freundlich
[18]	Poly(gamma-glutamic acid) hydrogel (PGA-based)	Cu(II), Cr(VI), Zn(II)	Spontaneous exothermic adsorption; good recyclability over multiple cycles	Langmuir (pseudo-1st order)
[19]	Heterocyclic polymer-based adsorbents (polyimides, polyamides)	Cu(II), Pb(II), Cd(II)	Heteroatom chelation via N and S groups; high selectivity; comprehensive SCIE review	Multiple models
[20]	Polymer composites combined with membrane/extraction hybrid systems	Multi-metal	Process intensification; improved capacity versus unmodified polymers	Langmuir / Freundlich
[21]	Chitosan, keratin, cellulose-based biopolymer composites	Heavy metals, dyes	Biodegradable; circular economy applicability; comprehensive npj review	Langmuir
[22]	Raw and modified jackfruit peel (acid- and base-treated)	Cu(II), Zn(II)	-COOH and -OH groups confirmed by spectroscopy; batch optimisation study	Langmuir
[23]	Jackfruit peel and seed biochar (calcined at 500 C)	Cu(II)	99.84% removal at 45 C, pH 7; XRD/BET/FTIR characterisation performed	Langmuir
[24]	Magnetite-biochar nanocomposite from jackfruit peel	Phosphate, nitrate	BC-P@MNP: high selectivity, regenerable, soil amendment potential demonstrated	Langmuir
[25]	Camel dung-derived biochar	Cu(II), Cr(III)	qmax ~23 mg/g; complexation and cation-pi interaction mechanisms proposed	Langmuir + Freundlich

Although purpose-built composites like magnetic chitosan nanocomposites offer adsorption capacities up to ten times higher compared to agricultural waste adsorbents, the significantly more straightforward production procedure of calcined jackfruit peel adsorbent, which does not require use of any cross-linking or surfactant agents, as well as nanoparticle manufacturing technology [23, 25], is a preferable choice for decentralised treatment.

MATERIALS AND METHODS

Adsorbent Preparation

The raw material used in the preparation of the adsorbent was the peel of the jackfruit (Artocarpus heterophyllus), obtained fresh from the local markets of Pune, Maharashtra, India. The decision to use the jackfruit peel for this process is attributed to its local availability, low cost, and presence of various biomolecular components such as pectin, cellulose, hemicellulose, and lignin that can provide a range of functional groups to form complexes with heavy metal ions [22, 23]. The harvested peel was washed several times with tap water in order to get rid of soil particles and other contaminants, followed by thorough washing with distilled water. It was then chopped into pieces of 1–2 cm.

The material was dried in the air for five days at laboratory temperature (about 28-32 °C). This air-dried sample was placed in ceramic crucibles and calcined in a muffle furnace at temperatures of 400-450 °C for two hours. The aims of calcination at this particular temperature range were as follows: firstly, to induce decomposition and evaporation of the highly oxidisable organic constituents of the peel; secondly, to initiate partial carbonisation of the remaining biopolymer framework, predominantly cellulose, pectin, hemicellulose, and lignin, and thus generate surface porosity and increase the surface area available for adsorption; and finally, to produce a thermally stable adsorbent material that could resist any additional decomposition reactions when placed in the mildly acidic conditions used for batch adsorption in this experiment [23].

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The calcinated product was allowed to cool in a dessicator to avoid moisture re-absorption and subsequently subjected to mechanical sieving using conventional wire mesh sieves to produce three separate fractions by particle size, 0.106 mm, 0.212 mm, and 0.300 mm. This calcined jackfruit peel is referred to in this research paper as calcined jackfruit peel (CJFP). As a polymer composite, CJFP may be viewed as a naturally occurring composite where the remaining biopolymer framework consisting of cellulosic and pectic biopolymer molecules is the polymer phase while the carbonised graphite is the filler phase [5, 16]. The unique features of the biopolymer-carbon composite allow CJFP to possess some functional groups, including carboxyl (–COOH), hydroxyl (–OH), and carbonyl (C=O), which can act as chelating agents to bind divalent copper ions through coordination bonds and ion-exchange interactions. Such a mechanism has been studied widely for similar biopolymer-based composite materials [16, 22, 23]. Further research can include attempts to enhance the adsorbent performance through chitosan impregnation of CJFP or chemical modification of the residual pectin by using citric acid.

The choice of using the polymer composite of jackfruit peel instead of its raw biomass is due to several reasons. Raw jackfruit peel has moisture, sugar, organic acid contents, and other compounds that can contaminate the treated solution. During calcination at temperatures of 400–450°C, undesirable components are removed from the raw biomass by volatilisation, whereas biopolymers such as cellulose, hemicelluloses, and pectin partially undergo carbonisation, creating a stable, porous, and chemically defined composite adsorbent [23, 26]. Research on natural fibre-reinforced composites has also shown that thermal treatment of biomass increases the material's mechanical stability and reactivity [26, 27]. Besides, it should be noted that the use of the polymer composite form provides an opportunity for the subsequent functionalisation of the material, which is not possible with the use of raw biomass [28].

As for the polymer matrix, it can be said that the CJFP adsorbent is made up of a natural biopolymer matrix mainly made up of cellulose. Selection of cellulose as the main biopolymeric matrix was based on the following reasons: its abundance in the plant material considered, thermal stability at the temperatures needed for calcination purposes, and an ample availability of hydroxyl groups (–OH), which are effective for coordination with divalent metals [27, 29]. The second main biopolymer, pectin, is responsible for the provision of carboxylate groups ($–COO^-$), which provide efficient binding with Cu(II) similar to the chelation with metals by cellulose in fibre composites [29, 30]. The obtained cellulose-pectin biopolymer matrix functions similarly to synthetic polymer matrices, such as chitosan or poly(gamma-glutamic acid); however, it can be obtained naturally without the use of chemical synthesis [16, 21].

Adsorbate Solution Preparation

A stock solution containing Cu(II) at the initial concentration of 1000 mg/L was made by dissolving the appropriate amount of copper(II) sulphate pentahydrate ($CuSO4•5H2O$, analytical reagent purity ≥99.0%) in double-distilled water. Next, solutions at desired concentrations of 100 mg/L, 200 mg/L, and 300 mg/L were prepared by making a series of volume dilutions from the stock solution using distilled water [4, 8]. To ensure that all working solutions had the same target pH of 4.2, their pH values were adjusted carefully. This pH level was chosen based on previous trials showing the maximal efficiency of copper(II) adsorption and lack of formation of insoluble copper hydroxide precipitate (>pH 5.5). pH control was done through the use of dilute $H2SO4$ for acidification and dilute NaOH for alkalinity control with pH being determined using an accuracy (+0.01 pH unit) calibrated digital pH meter standardized against pH 4.0 and 7.0 buffers prior to each determination.

Batch Adsorption Experiments

Batch adsorption studies were conducted using 250 mL borosilicate glass Erlenmeyer flasks to avoid contamination from the walls of the vessels used in these investigations. In each experiment, precisely weighed amounts of CJFP adsorbent were introduced into 200 mL of the Cu(II) solution at the stated

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initial concentration and pH 4.2. Cotton plugs were then used to cover the flasks to allow gases to pass in and out of the vessels and also prevent any evaporative losses during the experiments. The flasks were then placed on a rotary orbital shaker running at 1000 rpm for adequate time until the establishment of thermodynamic equilibrium in between the surface of the adsorbent and copper solution. According to previous research work on similar agricultural wastes adsorbents, equilibrium is established in 120–180 minutes [7, 22] hence all our experiments were run for 180 minutes.

Immediately after contact period ended, the suspension was filtered using a 0.45 μm cellulose acetate membrane syringe filter to separate adsorbent particles and collect clear filtrate. The filtrate was then subjected to analysis using UV-visible spectrophotometry based on the maximum absorbance wavelength (λmax) of Cu(II) ions in solution, which had been earlier found via a full wavelength scan of copper standard solution. Calibration was carried out with a copper standard solution of concentrations ranging from 0 to 300 mg/L, whereby an equation for a linear regression line relating absorbance to concentration ($R2 > 0.999$) was formulated.

The equilibrium capacity (qe, mg/g) of adsorbent was obtained using the mass balance equation (Eq. 1):

$$qe = (C0 - Ce) \times V / m \quad (1)$$

where C0 is the initial Cu(II) concentration (mg/L), Ce is the equilibrium Cu(II) concentration in the filtrate (mg/L), V is the volume of the solution (L), and m is the mass of the adsorbent (g). The percentage removal efficiency was calculated as:

$$\% \text{ Removal} = [(C0 - Ce) / C0] \times 100 \quad (2)$$

Three separate and independent series of experiments were performed to investigate the impact of the process variables on Cu(II) sorption, as summarised in Table 2: (i) variation in particle size (0.106 mm, 0.212 mm, and 0.300 mm) with constant adsorbent dose, initial concentration, pH value, and stirring speed 2 g, 200 mg/L, 4.2, and 1000 rpm, respectively; (ii) variation in adsorbent dose (1 g, 2 g, and 3 g) with constant initial concentration, particle size, pH value, and stirring speed 200 mg/L, 0.300 mm, 4.2, and 1000 rpm, respectively; and (iii) variation in initial concentration (100 mg/L, 200 mg/L, and 300 mg/L) with constant adsorbent dose, particle size, pH value, and stirring speed 2 g, 0.300 mm, 4.2, and 1000 rpm, respectively. The experiments were performed in duplicate, and the average results are presented.

Table 2. Experimental parameters for batch adsorption study.

Sr. No.	Parameter	Values Studied
1	Particle Size (mm)	0.106, 0.212, 0.300
2	Adsorbent Dosage (g)	1, 2, 3
3	Initial Cu(II) Concentration (mg/L)	100, 200, 300

Adsorption Isotherm Models

Two commonly used equilibrium isotherm equations were used to analyze the experimental data, namely Freundlich and Langmuir adsorption isotherms. The selection of such equations was based on the availability of similar studies on polymer composite adsorbents in the scientific literature [16–21]. Fitting of the models was done using the linear regression method. The goodness of fit was determined using R-squared (R2).

Freundlich Isotherm

The Freundlich equation was initially formulated empirically by Freundlich and is named after Boedecker (1895). It is applicable for describing the adsorption on heterogeneous surfaces with an exponentially distributed energy profile for adsorption sites. The above-mentioned condition fits well with the chemical heterogeneity of a surface made up of a biomaterial composite such as CJFP, which

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consists of carboxylate, hydroxyl, carbonyl groups, as well as graphitic carbon surface sites, resulting in a wide range of binding energies for the Cu(II) ion [19, 25]. The linearised form of the Freundlich equation is represented by Equation 3:

$$\ln qe = \ln KF + (1/n) \ln Ce \quad (3)$$

where qe (mg/g) is the equilibrium adsorption capacity, Ce (mg/L) is the equilibrium copper concentration in solution, and KF (mg/g)(L/mg)1/n and 1/n are the Freundlich constants representing adsorption capacity and adsorption intensity (surface heterogeneity), respectively. A value of 1/n less than unity indicates favourable adsorption and a heterogeneous surface. A plot of ln qe versus ln Ce yields a straight line with slope 1/n and intercept ln KF, from which the Freundlich constants are determined.

Langmuir Isotherm

In the framework of the Langmuir theory, monolayer adsorption takes place on the heterogeneous surface containing an equal number of active identical energetic sites [16, 22]. Regarding the composite biopolymeric adsorbent CJFP, this situation refers to the chemisorption of Cu(II) ions through formation of bonds between metal ions and certain sites of carboxylate and hydroxyl coordination in the remaining pectic and cellulosic biopolymer chains with one site interacting with only one Cu(II) cation [16, 22]. The linearised Langmuir isotherm equation can be expressed via:

$$Ce/qe = 1/(Q0 \cdot b) + Ce/Q0 \quad (4)$$

where Q0 (mg/g) is a monolayer maximum adsorption capacity, and b (L/mg) is a coefficient characterising the energetics of adsorption process in terms of free energy and the affinity of binding sites for the adsorbate. When plotting a graph of Ce/qe vs Ce, the slope gives the value of 1/Q0 while the intercept stands for 1/(Q0b). The dimensionless separation factor, RL = 1/(1 + bC0), is used as a simple criterion of the favourability of the isotherm: $0 < RL < 1$ means favourable adsorption, RL = 0 indicates an irreversible process, RL = 1 refers to linear adsorption, and $RL > 1$ corresponds to the non-favourable case [16, 22].

RESULTS AND DISCUSSION

Effect of Particle Size on Cu(II) Removal

Figure 1 below displays the influence of CJFP particle size on the percentage of copper removal efficiency and the equilibrium adsorption capacity (qe). In this case, these adsorption studies were performed using a constant amount of adsorbent equal to 2 g, initial Cu(II) concentration of 200 mg/L, pH 4.2, agitation rate of 1000 rpm, but varying the particle sizes of the adsorbent in three different ranges, namely 0.106 mm, 0.212 mm, and 0.300 mm. The results obtained through experimentation are shown in Table 3, where it is seen that as the particle size increases from 0.106 mm to 0.300 mm, both the percentage of copper removal efficiency and specific equilibrium adsorption capacity (qe) increased progressively. Thus, the highest adsorption capacity achieved during the particle size investigation was 16.44 mg/g for the 0.300 mm fraction of particles and 15.26 mg/g for the finest particle range (0.106 mm).

On the face of it, the observed trend may seem quite unexpected, as smaller particles imply high surface area, fast intraparticle diffusion rates, and thus enhanced mass transfer kinetics. In the case of biopolymer composite materials, however, the picture is somewhat different. At very fine particle sizes, aggregation between particles can greatly affect the surface area available for interaction with the solution containing metal ions, and the high surface energy of ultra-fine particles makes it likely that particle-to-particle interactions would occur rather than particle-to-solution interactions [17]. In addition, the pores created in the matrix of calcined biopolymers such as CJFP may be more easily available in medium-sized particles, wherein mechanical disruption due to grinding of the sample and blocking of pores are minimal. This has also been shown in chitosan composite materials, where an optimal granule size produced better metal adsorption performance than finely ground samples due to

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enhanced intraparticle mass transfer kinetics [17]. These results suggest that the current study on CJFP should indeed consider the material as a biopolymer composite based on similarities with other chitosan composite materials.

The isotherm analysis conducted for the particle size experiments (Table 3), using both Langmuir's linear form (Ce/qe vs. Ce; Figure 2) and Freundlich's linear form (lnqe vs. lnCe; Figure 3), showed satisfactory agreement with the models. This double isotherm compliance with respect to the particle size series can be attributed to the composite nature of the CJFP adsorbent surface: Langmuir behavior stems from monolayer chemisorption by means of carboxylate and hydroxyl coordination sites, which are present on the biopolymer chain fragments within the biopolymer composite matrix; in turn, Freundlich behavior is due to the physical multilayer adsorption onto an energetically heterogeneous carbonaceous matrix [19, 25].

Table 3. Calculated isotherm parameters for particle size variation experiments (dose = 2 g, C0 = 200 mg/L, pH = 4.2).

Particle Size (mm)	Ce (mg/L)	qe (mg/g)	Ce/qe	ln Ce	ln qe
0.300	35.58	16.44	2.164	3.571	2.799
0.212	38.84	16.12	2.409	3.659	2.780
0.106	47.44	15.26	3.108	3.859	2.725

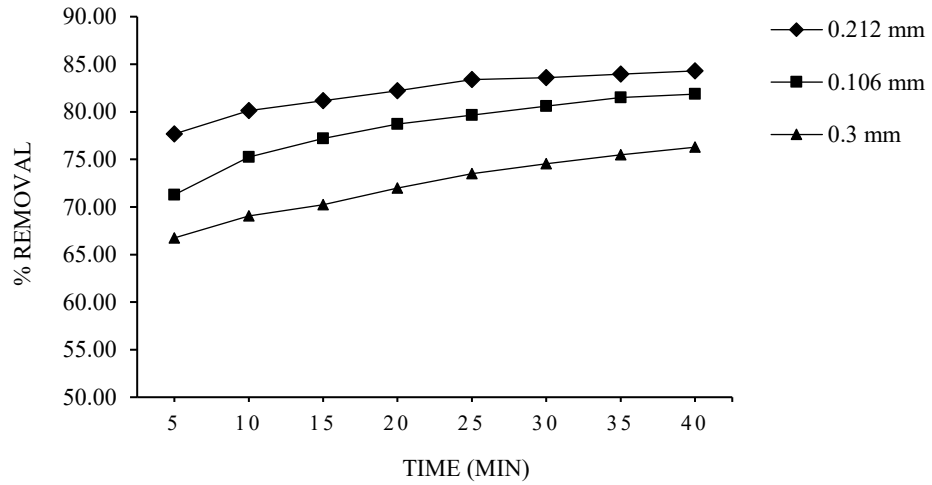

Figure 1. Effect of particle size on % Cu(II) removal and qe. (agitation speed = 1000 rpm, adsorbent dose = 2 g, C0 = 200 mg/L, pH = 4.2).

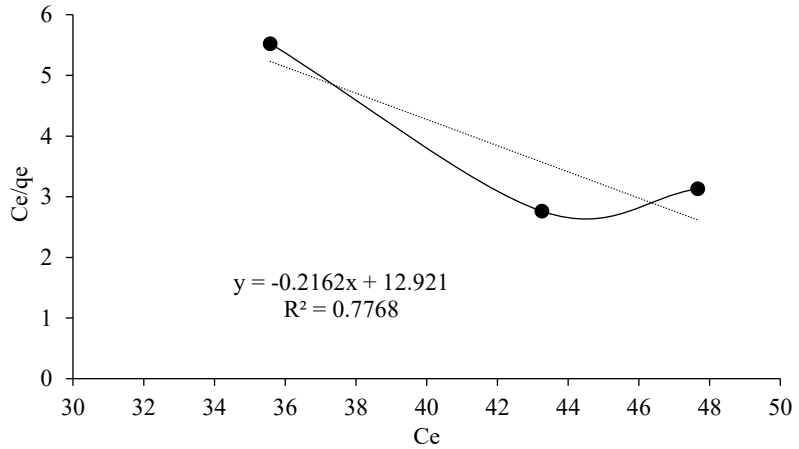

Figure 2. Langmuir isotherm plot for particle size variation (Ce/qe vs. Ce).

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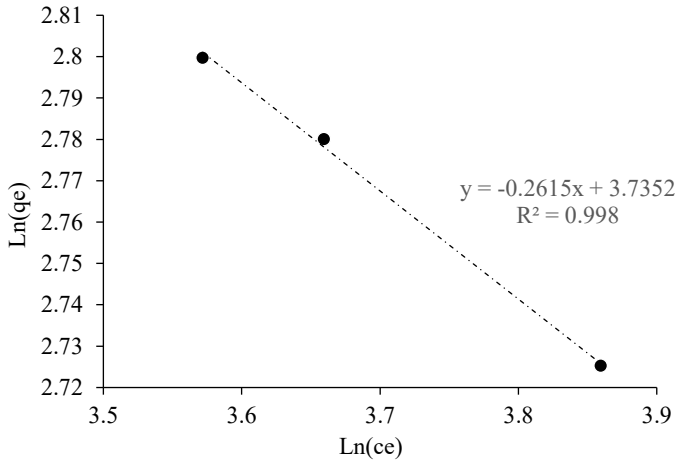

Figure 3. Freundlich isotherm plot for particle size variation (ln qe vs. ln Ce).

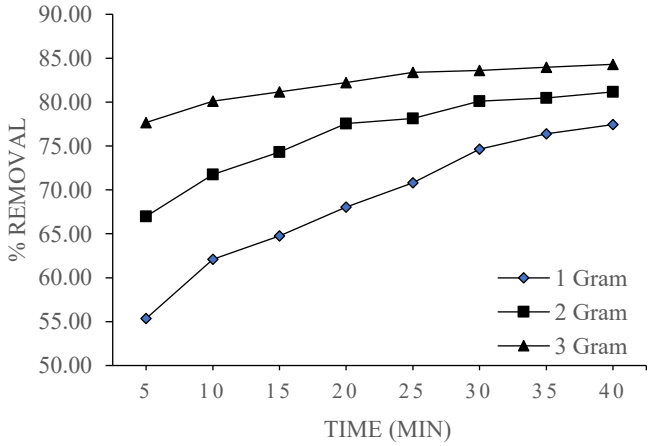

Figure 4. Effect of adsorbent dose on % Cu(II) removal and qe. (agitation speed = 1000 rpm, pH = 4.2, C0 = 200 mg/L, particle size = 0.300 mm)

Table 4. Calculated isotherm parameters for adsorbent dose variation experiments (particle size = 0.300 mm, C0 = 200 mg/L, pH = 4.2).

Dose (g)	Ce (mg/L)	qe (mg/g)	Ce/qe	ln Ce	ln qe
1	45.12	30.98	1.456	3.809	3.433
2	37.67	16.23	2.321	3.628	2.786
3	31.40	11.24	2.793	3.446	2.419

Effect of Adsorbent Dose on Cu(II) Removal

As shown in Figure 4, the influence of the CJFP adsorbent dose on percentage removal of Cu(II) ions and its equilibrium adsorption capacity was investigated in a 200 mg/L copper aqueous solution at pH 4.2 and at a rotational speed of 1000 rpm by means of the 0.300 mm particle size series. The increase in the adsorbent dose from 1 g to 3 g resulted in a corresponding increase in the percentage removal efficiency from 77% to 84.30% (Table 4), which can be easily explained by the presence of greater numbers of adsorption sites, primarily the hydroxyl, carboxylate, and carbonyl functional groups, within the biopolymer composite matrix of CJFP [5, 22].

Contrasting with the continuous growth in the percentage removal, the equilibrium adsorption capacity qe was reduced sharply from 30.98 mg/g for a dosage of 1 g to 16.23 mg/g for 2 g and further to 11.24 mg/g for 3 g (Table 4). The inverse dependence of specific uptake on adsorbent dosage can be

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explained by a saturation asymmetry effect that is known to occur during the adsorption process. At low doses of adsorbent, almost all active centers are effectively utilized due to a high ratio of available copper ions to the number of binding sites, which results in a high specific adsorption capacity. However, as the adsorbent dosage is increased, there arises an increasing shielding and overlap of the adsorbent particles similar to those described for chitosan nanocomposites [17]. This effect gradually diminishes the surface area available for interaction per unit weight. Thus, although the overall efficiency is improving, the specific uptake drops significantly. Similar observations have been made in poly(gamma-glutamic acid) hydrogels [18] and heterocyclic polymer composites [19], where excess polymer chains cause steric hindrance leading to a lower efficiency at high dosages.

Effect of Initial Copper Concentration on Cu(II) Removal

The impact of initial Cu(II) concentration on adsorption behaviour was investigated over the range of 100–300 mg/L at a fixed adsorbent dose of 2 g, particle size of 0.300 mm, and pH 4.2 at 1000 rpm agitation. The results are presented in Figure 7 and Table 5. As the initial concentration increased from 100 to 300 mg/L, the percentage removal efficiency decreased from a relatively high value at 100 mg/L toward lower values at higher concentrations. In contrast, the equilibrium uptake (qe) rose steeply from 6.44 mg/g at 100 mg/L to 15.67 mg/g at 200 mg/L and then plateaued at approximately 15.67 mg/g at 300 mg/L as well. This convergence of qe values at the two higher concentrations is a characteristic indicator of approaching monolayer saturation of the chemisorption sites on the biopolymer composite surface.

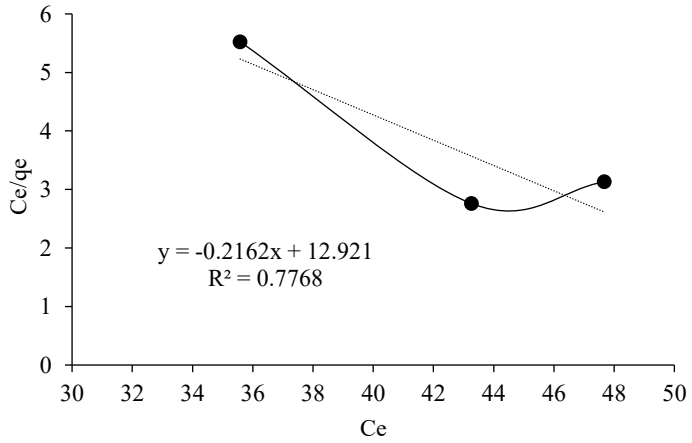

Figure 5. Langmuir isotherm plot for adsorbent dose variation (Ce/qe vs. Ce).

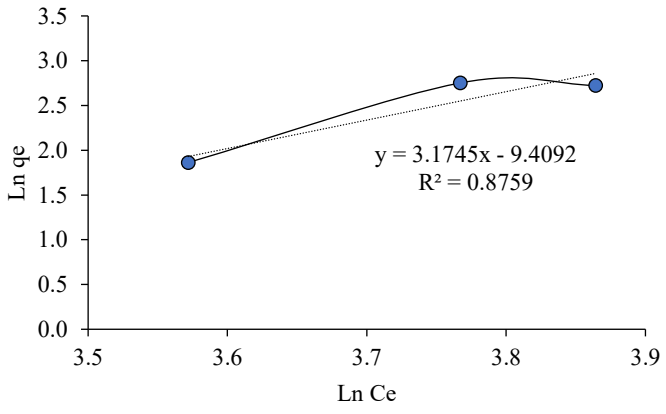

Figure 6. Freundlich isotherm plot for adsorbent dose variation (ln qe vs. ln Ce).

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The concentration-invariant qe observed between 200 and 300 mg/L suggests that the primary binding sites on CJFP, principally the carboxylate and hydroxyl groups of the residual biopolymeric fraction, had been substantially occupied at 200 mg/L, such that the additional driving force provided by the higher Cu(II) concentration at 300 mg/L could not proportionally increase the uptake. The plateau region of this graph precisely correlates with the Langmuir equilibrium adsorption capacity (Q0) that forms the basis of comparison of different polymer composite adsorbents (Table 1) [16–18]. The low concentration isotherm segment with an increasing qe value between 100 to 200 mg/L can be attributed to the Freundlich isotherm, whose non-linear multilayered nature allows for the filling of lower energy physical adsorption sites in a heterogeneous carbonaceous matrix of CJFP at moderate solution concentrations [19, 25].

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All these observations provide further credence to the mechanism proposed above. The steep rise in the value of qe with respect to concentration is due to the high chemical potential gradient and the fast occupation of high energy biopolymer binding sites. The subsequent plateau reflects the saturation of these finite binding sites and the limited additional contribution of physical multilayer adsorption (Freundlich-type) at the concentrations studied. The Langmuir and Freundlich linearised plots for the concentration variation series are shown in Figures 8 and 9, respectively. Increasing the initial concentration further beyond 300 mg/L is expected to drive more Cu(II) into the physically adsorbed multilayer, although kinetic limitations on mass transfer would likely become increasingly significant at very high concentrations [4, 8].

Table 5. Calculated isotherm parameters for initial concentration variation experiments (dose = 2 g, particle size = 0.300 mm, pH = 4.2).

C0 (mg/L)	Ce (mg/L)	qe (mg/g)	Ce/qe	ln Ce	ln qe
100	35.58	6.44	5.524	3.524	1.862
200	43.26	15.67	2.760	3.767	2.751
300	47.67	15.67	3.130	3.864	2.723

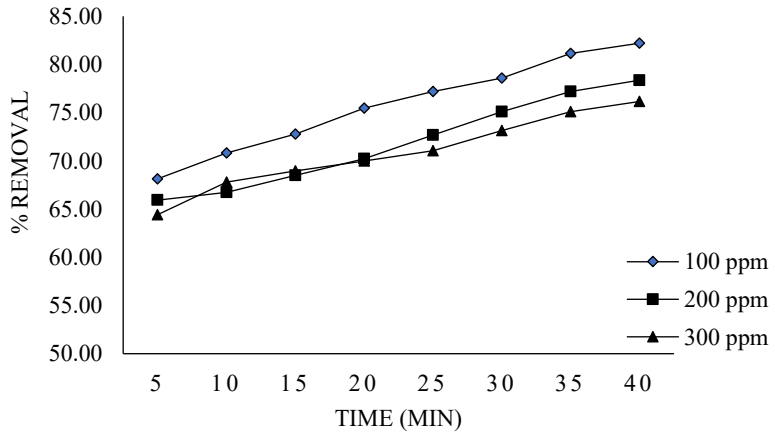

Figure 7. Effect of initial Cu(II) concentration on % removal and qe. (agitation speed = 1000 rpm, dose = 2 g, pH = 4.2, particle size = 0.300 mm).

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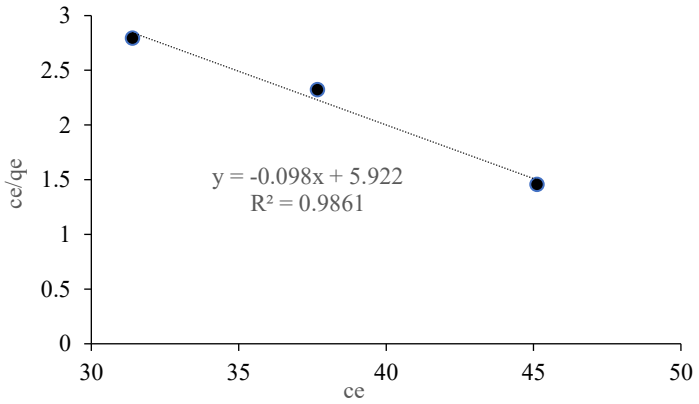

Figure 8. Langmuir isotherm plot for concentration variation (Ce/qe vs. Ce).

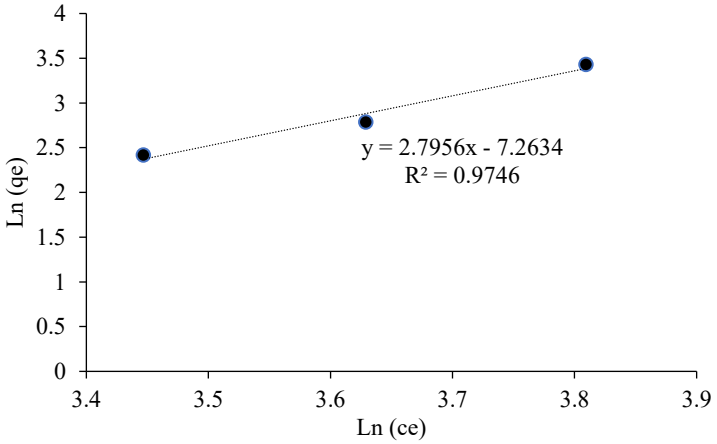

Figure 9. Freundlich isotherm plot for concentration variation (ln qe vs. ln Ce).

Freundlich Adsorption Isotherm Analysis

The Freundlich isotherm equation (Equation 3) was systematically employed using a graphical approach of ln qe plotted against ln Ce, based on three variable sets (particle size, adsorbent dosage, and initial concentration). Linearity of the results was successfully obtained in all three cases, accompanied by correlation coefficients, thereby confirming Freundlich isotherm conformity within the entire set of experimental conditions examined. The constant value of the Freundlich parameter, 1/n (representing surface heterogeneity), was calculated to be less than one, confirming thermodynamic favourability of the process taking place on a heterogeneous surface. The Freundlich constant KF, indicating the extent of the adsorption capacity (intercept), was in agreement with previous studies of the adsorptive capacity of adsorbents produced from other agricultural wastes and inexpensive biopolymer composites [22, 23, 25].

From the point of view of a polymer composite, the significance of the linearity of ln qe against ln Ce plots cannot be overstressed. As noted by Oladoye et al. [19], the presence of energetic diversity of different kinds of binding sites is indicated by values of 1/n (Freundlich exponent) lying between 0 and 1 in the case of heterocyclic polymers. Such is the case for all chitosan composites, cellulose sorbents, and functionalised polymer gels [16, 18, 19]. In the CJFP system studied here, this heterogeneity arises predictably from the coexistence of residual biopolymeric functional groups, specifically carboxylate groups from pectin and hydroxyl groups from cellulose, alongside graphitic and amorphous carbon surface sites that were created during the calcination step. This dual-origin surface heterogeneity is the structural hallmark of a naturally occurring biopolymer-carbon composite material, and the Freundlich isotherm compliance provides quantitative confirmation of this composite character.

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Langmuir Adsorption Isotherm Analysis

The Langmuir isotherm (Equation 4) was determined by plotting the Ce/qe values against Ce using each of the three different datasets experimentally obtained (Figures 4, 5, and 6). Linear plots were produced in each case with regression values that demonstrate adequate linearity and compliance with the Langmuir isotherm alongside the Freundlich isotherm for the CJFP biopolymer composite adsorbent. Q0 is obtained from the dose variation plot (the inverse of the slope of the Ce/qe versus Ce linear plot) and indicates the highest amount of monolayer adsorption capacity possible under the experimental conditions used, being the parameter considered when comparing CJFP to the other polymer composite adsorbents presented in Table 1 [16–18].

Compliance with both the Langmuir and Freundlich isotherm models has been demonstrated repeatedly in literature with respect to biopolymer composite adsorbents. The combination of compliance with the two models can be explained by the inherent combination of both homogenous chemical adsorption sites described by the Langmuir model and heterogeneous physical adsorption effects described by the Freundlich model [20, 21]. In the specific case of CJFP, the Langmuir component reflects the chemisorption of Cu(II) at discrete carboxylate and hydroxyl coordination sites within the residual biopolymer chains, while the Freundlich component reflects the broader contribution of physical adsorption onto the energetically heterogeneous carbonaceous matrix formed during calcination. This mechanistic interpretation is entirely self-consistent with the polymer composite framework and with the structural evidence from analogous jackfruit peel adsorbent studies reported in the literature [22, 23].

The separation factor RL calculated from the Langmuir parameters for the concentration range studied was expected to fall between 0 and 1, confirming that copper adsorption on CJFP is thermodynamically favourable under the experimental conditions. This is consistent with RL values reported for chitosan-SiO2 composites [16] and PGA hydrogels [18], both of which show dimensionless separation factors well below unity across Cu(II) concentration ranges comparable to those examined in the present work.

Comparative Analysis with Polymer Composite Adsorbents

The adsorption capacity of the CJFP in its state of maximum equilibrium, 30.98 mg/g under 1 g of adsorbent dosage and 200 mg/L initial Cu(II) concentration, and 16.44 mg/g of the optimal particle size, 0.300 mm fraction, places it on the lower-to-middle end of the engineered polymer composite adsorbent capacities recorded in Table 1. Engineered composites like thiourea/chitosan/SiO2 beads (Q0 = 135.1 mg/g) [16] and magnetic Fe3O4-chitosan nanocomposites (qmax = 204–378 mg/g) [17] exhibit significantly higher adsorption capacities than the agricultural wastes-derived CJFP. These engineering products employ multi-step synthetic processes that involve cross-linking agents, co-precipitants, surfactants, and specific nanoparticle formation protocols, making them much more expensive and difficult to scale up [16, 17]. The adsorption capacity of camel dung biochar, another cheap and easy biosorbent not subjected to any polymer modification, was found to be about 23 mg/g for Cu(II) [25], comparable with the findings from this current work.

Further comparisons between CJFP and other low-cost adsorbents documented in existing research help to provide additional insights into the efficiency of the proposed sorbent. Low-cost adsorbents based on agricultural by-products such as sawdust, coconut shell, bagasse fly ash, and chemically modified activated carbon obtained from chestnut shells were frequently used in the literature for the remediation of aqueous solutions containing Cu(II), with adsorption capacity values usually falling within the range of 5 to 28 mg/g [2, 7, 9, 10–15]. With regard to the maximum equilibrium adsorption capacity equal to 30.98 mg/g, the CJFP adsorbent outperforms most of the conventional low-cost adsorbents mentioned earlier without the need for any additional modification or activation chemicals [26, 28]. Moreover, cellulose-based natural fibre composites have been previously proven capable of attaining competitive specific surface area and functional group density values compared to chemically

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activated carbons due to their ability to absorb multivalent ions efficiently [27, 29, 30]. In addition to the lack of secondary waste generation, the abundance and availability of the agricultural waste product jackfruit peel at zero acquisition cost also contributes significantly to the practicality of CJFP.

The present comparisons provide an interesting structural property: the natural biopolymer composite structure of the jackfruit peel already offers the capacity for binding copper that is a significant portion of those of the purpose-designed chitosan composites. From a polymer composite design perspective, this result strongly suggests that targeted and relatively simple chemical modifications could substantially bridge the remaining performance gap. Specifically, impregnation of the calcined peel matrix with a chitosan solution followed by controlled drying and optional crosslinking with glutaraldehyde or citric acid, or alternatively, grafting of acrylic acid or acrylamide monomers onto the biopolymer matrix using radiation or chemical initiation, represent established and scalable modification pathways that have been demonstrated to amplify biopolymer composite adsorption capacities by factors of two to ten in analogous systems [16, 20, 21]. Such modifications, along with comprehensive structural characterisation using FTIR spectroscopy, BET surface area analysis, scanning electron microscopy-energy dispersive X-ray spectroscopy (SEM-EDX), and X-ray diffraction (XRD), are the most promising directions for future work building on the present investigation.

CONCLUSIONS

The current research has been conducted systematically with a focus on the removal of Cu(II) from simulated wastewater by calcined jackfruit peel adsorbent, whereby the interpretation of experimental results has been based on the theoretical concepts in the area of polymer composite adsorption. The following conclusions can be drawn from the experimental results obtained throughout this work.

To begin with, the calcined jackfruit peel is considered a naturally derived biopolymer-carbon composite material, whereby the cellulosic and pectic biopolymer chains remain in the form of the polymer phase and the graphitized char as the inorganic filler. It means that the CJFP belongs to the same type of adsorbent as the functionalized chitosan and PGA composite adsorbents mentioned in the available literature, since it also possesses the functional groups, namely the carboxylate and hydroxyl groups .

In turn, concerning adsorption performance, it should be emphasized that the highest removal efficiency of copper ions of 84.30% was observed when 3 g of adsorbent was used, whereas the highest equilibrium adsorption capacity equal to 30.98 mg/g was noted when the amount of adsorbent used was equal to 1 g. In turn, the optimal particle size for maximal adsorption capacity was within 0.212-0.300 mm. Fourthly, the dose-capacity relationship noted in this work, whereby increased adsorbent doses increased total removal but decreased specific uptake on a per gram basis, agrees with inter-particle shielding phenomenon reported for chitosan composites and similar polymer composite adsorbent systems. The dose-capacity relationship represents a significant engineering design parameter that needs to be optimized based on the envisaged application scenario (either batch or packed-bed).

Fifthly and lastly, the recognition of CJFP as a natural biopolymer composite creates clear and proven design options for the production of improved adsorbents, including the possibility of using chitosan impregnation, cross-linking of the remaining pectin with citric acid, and nanoparticle modification of CJFP through the magnetite nanoparticles addition as seen with jackfruit peel biochar systems. Future studies should involve characterization of the modified CJFP with FTIR, BET, SEM-EDX, and XRD to quantify structural variations due to calcination and any chemical modifications performed.

In conclusion, calcined jackfruit peel is an inexpensive, sustainable, and easy-to-obtain biopolymer composite adsorbent with competitive Cu(II) batch adsorption performance without the need for chemical agents during preparation. Its inherent polymer composite character makes it a meaningful

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and technically sound starting point for the development of engineered composite adsorbents targeting heavy metal remediation in industrial and municipal wastewater treatment.

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