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Research Article

Physicochemical Assessment and Cu-Nano Hybrid Modification of Palm Fronds for Cadmium Adsorption: Kinetics and Thermodynamics

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ISSN : 2688-8394

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Submission: October 28, 2025
Published: March 05, 2026

Volume 5 - Issue 3

How to cite this article: Ikeh Obianuju Adaobi*, Ojiako Eugenia Nnonye, Nwadiogbu Joseph Onyebuchi, Ejidike Lynda Chinyere, Okonkwo Ngozi Anastasia and Nwankwo Njideka Veronica. Physicochemical Assessment and Cu-Nano Hybrid Modification of Palm Fronds for Cadmium Adsorption: Kinetics and Thermodynamics. Ann Chem SciRes. 5(3). ACSR. 000615. 2026.
DOI: 10.31031/ACSR.2026.05.000615

Abstract

Physicochemical evaluation of agricultural wastes underpins their energy potential, adsorption performance and environmental implications. This work aimed at assessment of the physical and chemical constituents of palm fronds and evaluation of raw (RPF) and copper nanocomposite-modified (CuPF) variants for cadmium ion removal from wastewater. Proximate (bulk/tapped density, moisture and ash contents, volatile matter) and ultimate (lignin, cellulose, hemicellulose, holocellulose) compositions, alongside water/oil absorption, were quantified via thermogravimetric analysis. Batch experiments optimized contact time, pH, dosage, temperature, and initial Cd ion concentration, with analysis by atomic absorption spectrometry (AAS). The results of the physicochemical properties showed that palm fronds are capable of providing internal volumes for the penetration and retention of metal ions. CuPF generally showed higher adsorption capacity (qe) to RPF across all conditions studied. Pseudo-second-order kinetic superseded, with intraparticle diffusion (non-zero intercepts) indicating hybrid surface/diffusion control. Equilibrium favored Freundlich isotherm from R2 values (0.8009 and 0.8259) for RPF and CuPF respectively, evidencing cooperative adsorption. Thermodynamic parameters showed positive enthalpy and entropy values, denoting an endothermic process with increased disorder at the metal ion interface, while negative ΔG0 values confirmed spontaneity of the process. Conclusively, CuPF achieved >80% removal efficiency, establishing Cu-nanohybrid palm fronds as a low-cost, sustainable sorbent for cadmium pollution remediation.

Keywords: Physicochemical assessment; Palm fronds; Cadmium adsorption; Copper nano composites; Kinetics; Thermodynamics; Equilibrium

Introduction

Accelerated urbanization and industrialization have led to increased waste generation, and environmental pollution, particularly from heavy metals like cadmium [1,2]. Agricultural intensification exacerbates this issue, as agro-wastes, fertilizers, pesticides, and additives accumulate in soils and aquatic systems, causing long term ecological harm [3,4]. Agricultural wastes are specifically the non- edible portions of plants derived from the production, distribution, marketing, and consumption of fruits, vegetables, meat, dairy, and related products [5]. A significant amount of agro wastes and their by-products are generated globally in vast quantities, and often discarded without valorization, thereby amplifying pollution. Oil palm (Elaeis guineensis) is believed to originate from West and Central Africa and also found in in Thailand, Malaysia and Indonesia [6,7]. Higher production of oil palm is cultivated in plantations across the humid tropics of Asia, Africa and the Americas, from where its products are exported to global markets [7]. While palm yields sustainable unsaturated fatty acids and

essential minerals (Mg, Fe, Zn, K), its fronds are routinely discarded as low- value wastes, posing disposal burdens when mismanaged [6,7]. Yet, repurposing such wastes as adsorbents for heavy metal remediation harnesses their potential, obviating disposal needs and promoting circular economy principles. Palm fronds, in particular, hold promise for adsorption technologies and bioenergy due to their favorable physical and chemical traits governing material quality, reactivity and transformation [8-10]. Key parameters include bulk/tapped density, moisture/ash content, volatile matter, water/oil absorption capacity and compositional fractions (lignin, cellulose, hemicellulose, holocellulose). These components provide high surface areas and functional groups (e.g. hydroxyl groups from cellulose/ hemicellulose), necessary for pollutant sequestration from wastewater. Such analyses elucidate energy potential, adsorption efficacy, and environmental impacts [11-15]. Leveraging agro-wastes as adsorbents offers multifaceted benefits: it mitigates indiscriminate dumping or incineration-major pollution sources, while cutting disposal costs, boosting sustainability, and rivaling costly activated carbons in capacity without high preparation/ maintenance expenses [9,16]. Abundant and metal-binding rich, agro-wastes like corn cobs, palm shells and peanut shells feature porous structures, functional groups, thermal stability, antibacterial properties and tunable surface areas for remediation [17,18]. Notably, they exhibit exceptional fluid absorption (> initial weight) and undergo natural biodegradation via biological, physical, chemical, or photochemical pathways [19]. This study conducts a comprehensive physicochemical evaluation of palm fronds by assessing bulk/tapped density, moisture/ash content, volatile matter, lignin, cellulose, hemicellulose, holocellulose, and water/ oil absorption capacities via gravimetric methods and preparation of Cu- nano hybrid to evaluate cadmium ion adsorption efficacy from aqueous solutions under optimized conditions (contact time, pH, dosage, temperature, concentration), quantified by atomic absorption spectrometry. To the best of our knowledge, few studies have integrated all these parameters for palm fronds, underscoring the novelty of our multifaceted approach.

Materials and Method

Sample collection/preparation

Palm fronds used in this research were collected from a bush at Adazi-ani in Anaocha Local Government Area of Anambra state, Nigeria. The dirty material (palm fronds) was washed thoroughly under running water, dried properly in sunlight for (5h for three days) and then left to dry at 65 °C in the oven. After reducing the size, No 20 and 25 British Standard Sieve (BSS Sieves) were used to sieve the materials and corked in clean plastic bottle prior to all analysis. All reagents were analytical grade and used without further purification [20]. The analysis was done at Graceland Laboratories, Igwebuike Street, Awka, Anambra State, Nigeria.

Synthesis of copper nanoparticles

Exactly 0.2M of copper solution was prepared from $CuCl_2.2H_2O$ by dissolving 10g of the salt with 300mL water in a round bottomed flask under continuous stirring for 20min. About I mL of CH_3COOH was added to the solution and stirred for additional 30min. At the same time, 4.8g (8M) NaOH was dissolved in 15mL water in a separate beaker. The 8M NaOH solution was then added dropwise to the first solution using a micropipette under vigorous stirring and maintained at pH of 11while stirring for another 2h at 60 °C. Following this, the mixture was centrifuged at 5000rpm for 30min. The resulting precipitate was filtered using Whatman paper and washed several times with methanol and distilled water to eliminate any unreacted particles reactants and organic impurities. Finally, the particles were dried in a hot air oven at 200 °C for 20min [21].

Nanoparticle impregnated biomass

The copper nanocomposites synthesized were mixed in distilled water with constant stirring using a magnetic stirrer. Then, approximately 8.0g of pulverized biomass, pre-treated with 0.1 M $HN0_3$, was added to the mixture and stirred vigorously with a magnetic stirrer for 8h after stirring, the mixture was left to settle for 30min and then centrifuged at 8000rpm for 2h. The resulting hybrid material was filtered and dried in an oven at 70 °C for 24h. Finally, it was ground thoroughly into a fine powder using a mortar and pestle and passed through a 100-micrometer mesh sieve [20].

Physicochemical analysis of palm fronds

Determination of bulk density (DB)

Five grams of the sample was weighed and transferred into a separate 50mL dry measuring cylinders. The volume occupied by the sample was noted as the bulk volume. Therefore, the bulk density was determined by dividing the mass of the material by the bulk volume according to [22].

$$\text{Bulk density} = [M/VB] \quad (1)$$

where M is the mass of the sample and VB is the bulk volume of the sample

Determination of tapped density (DTa)

The measuring cylinder containing 5g of the sample was then tapped on a wooden platform by dropping the cylinder from a height of 1inch at 2secs intervals until there was no observable change in volume. The volume occupied by the material was recorded as the tapped volume. The tapped density was determined using the expression:

$$\text{Tapped density (DTa)} = [M/VT] \quad (2)$$

where M is the mass of the sample and VT is the tapped volume of the sample.

Moisture content determination

Two grams of the powdered sample was weighed, transferred into petri dish and then oven-dried for 3h at 105 °C to a constant weight. The moisture content (%) was then computed based on the initial air-dried weight as reported by [3].

Ash determination

One (1g) of sample was placed in a clean crucible of known weight (W_1). The crucible was placed in a muffle furnace at 600 °C

for 5h. The crucible and content were cooled in a desiccator and weighed again (W_2) according to the procedure by [13].

$$\%Ash = \frac{(W_2 - W_1)}{1g} \times \frac{100}{1} \quad (3)$$

Where W_1 = weight of crucible (g). W_2 = weight of crucible and ash (g)

Determination of water holding capacity

One gram of the sample was weighed and evenly distributed over the surface of a 10cm Petri dish. The sample was placed in a large desiccator containing distilled water in its reservoir and the weight gained by the exposed sample at 24h interval was recorded. The amount of water sorbed was calculated from the weight difference.

$$[(W_2 - W_1)/W_1] \times 100 \quad (4)$$

W_1 is the weight of the sample before exposure and W_2 is the weight of the sample after exposure [23].

Determination of lignin content

Exactly 1g of the sample was weighed and put in 100mL beaker. About 20mL of 72% sulfuric acid was added drop by drop with constant stirring by a small glass rod. After complete disintegration, the reaction was allowed to stand and the beaker covered with watch glasses and left-over night at room temperature. The sample was then transferred quantitatively to 1L round bottom flask, diluted with 3%sulfuric acid, boiled for 4h under reflux. The lignin was filtered on an ash less filter and washed with hot distilled water till neutrality, then gravimetrically estimated and ignited at 85 °C for 45min. The weight of ash was subtracted to give the ash free lignin percent [24].

Determination of hemicellulose content

Exactly 1g of extracted dried sample was transferred into a 250mL Erlenmeyer flask. About 150mL of 20g NaOH was added. The mixture was boiled for 3.5h with distilled water. It was filtered after cooling through vacuum filtration and washed until neutral pH. The residue was dried to a constant weight at 105 °C in a convection oven. The difference between the sample weight before and after this treatment is the hemicellulose content (%w/w) of dry biomass [25].

Determination of cellulose content

This was obtained by adopting the expression used by [26].

Cellulose = 100% - (Hemicellulose + lignin) (5)

Holocellulose estimation

Holocellulose is the total carbohydrate fraction (cellulose and hemicellulose) of the sample material.

Adsorption experiment

Cadmium salt $Cd(NO_3)_2$ was used to prepare solutions of lower concentrations (100-300mg/L). The effects of time, pH, concentrations, dosage and temperature on the adsorbents was monitored and the concentrations determined using Atomic Absorption spectrophotometer (PG- AA500F model manufactured by PG instruments Ltd). Other operating factors were kept constant, while changing the respective studied factor of interest. The uptake capacity qe(mg/g) was calculated from the mass balance equation below:

qe(mg/g) = (Co-Ce) V/m (6)

qe(mg/g) = equilibrium adsorption capacity, representing the amount of adsorbate per unit mass of adsorbent.

Co and Ce(mg/L) = initial and final concentrations of the adsorbate in the solution.

V = volume of the solution

M = mass of the adsorbent [16].

Results and Discussion

The result of the physicochemical constituents of palm fronds is presented in Table 1 below.

Table 1: Physicochemical compositions of the sample.

Parameters	Values
Ash content (%)	5.243
Volatile matter (%)	40.728
Moisture content (%)	5.9680
Bulk density(g/cm3)	0.3194
Tap density (g/cm3)	0.3407
Lignin (%)	17.000
Cellulose (%)	55.000
Hemi cellulose	28.000
Holo Cellulose	83.000
Water absorption capacity (g/g)	3.858
Oil absorption capacity (g/g)	3.301

The physical and chemical properties such as moisture and ash contents, volatile matter, cellulose and lignin of agro-waste materials play a vital role in determining their adsorption efficiency and selectivity. Effects of physicochemical parameters on the adsorption capacities of agro-waste materials are considered essential for evaluation of the adsorbents for adsorption of pollutants in aqueous solution. The moisture content of palm fronds was measured at 5.9680%. Low percentage of moisture would likely inhibit the activities of microorganisms and good for storage [27]. Bulk density is influenced by how particles arrange themselves and this arrangement changes as the powder is compressed and settles. It was observed that powders with finer particles and lower moisture levels tend to have a higher bulk density. On the other hand, tapped density also measures how well a powder can be packed into a confined space on repeated tapping. Values of bulk and tapped densities obtained in this study is within the range reported for energy usage [26]. The results obtained showed that ash content of palm fronds was (5.243%), suggesting a low level of inorganic residues. This is advantageous for adsorption process as high ash content often reduces porosity and sorbent effectiveness

[27]. Ash contents of this agro-waste material are close to the range used for energy and paper pulp applications as reported by [25,13]. Palm fronds belong to the lignocellulosic biomass group due to high lignin and cellulose contents that provides functional groups (hydroxyl groups) and surface area necessary to enhance adsorption process. The characterization of the morphological and structural properties of palm fronds was reported in previous research by the authors. Palm fronds can be utilized as adsorbents to remediate environmental pollution [28]. The values obtained in this research is in line with report from literature [25,13].

Effects of process parameters on the adsorption of cadmium onto palm fronds

Effects of time on the adsorption of cadmium onto palm fronds

Figure 1 shows the result of the effect of time on the adsorption of cadmium onto raw palm fronds (RPF) and Cu nano-composites of palm fronds (CuPF).

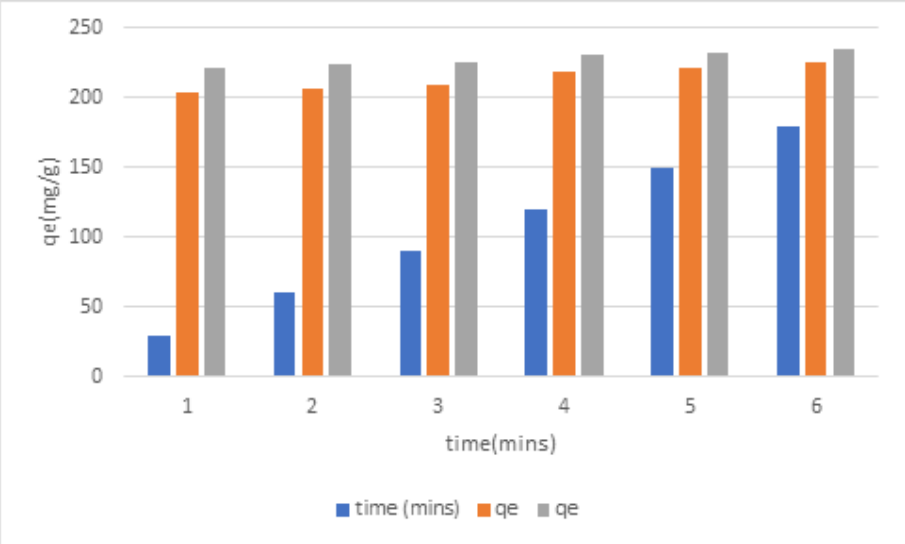

Figure 1: Plots for the effects of time on the adsorption of cadmium onto palm fronds.

It was observed that as sorption time increased from 30mins to 180mins, the maximum adsorption capacity (qe) increased as well. Throughout all the adsorption durations examined, CuPF had higher adsorption capacity compared to RPF, likely due to the increased number of active sites provided by the presence of copper molecules. These results align with previous studies reported in the literature [16,19]. According to a one-way ANOVA used to test for statistical significance of the process, CuPF are more efficient than that of RPF. It was observed that there is statistically significant difference in the mean efficiency of RPF and CuPF as shown in Table 3.

Effect of pH on the adsorption of cadmium onto palm fronds

The effect of solution pH on the adsorption of cadmium onto RPF and CuPF is shown in Figure 2.

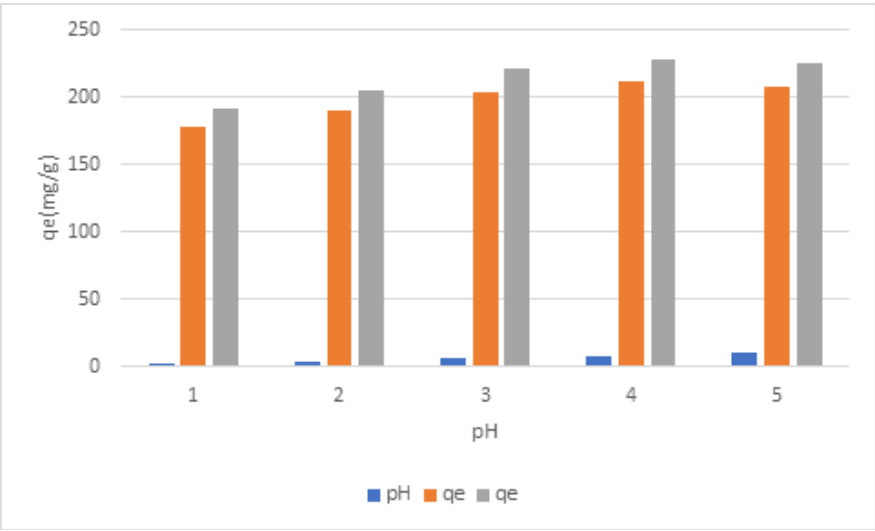

Figure 2: Plots for the effects of pH on the adsorption of cadmium onto palm fronds.

The equilibrium adsorption capacities were minimum at pH 2(178.6512mg/g for RPF and 191.5178mg/g for CuPF) and attained and became maximum at pH 8 (212.0465mg/g and 228.4019mg/g) for RPF and CuPF respectively. The decreased adsorption capacity at acidic pH is likely due to the competition between hydrogen ions (H+) and cadmium ions (Cd^{2+}) for the binding sites on the adsorbent, which reduces cadmium uptake. The finding was similar to studies conducted elsewhere [29]. A one-way ANOVA used to test for its statistical significance showed no significant difference in the mean efficiency of RPF and CuPF. meaning that the efficiency of raw and nano composites of adsorbents is relatively the same.

Effects of concentration on the adsorption of cadmium onto palm fronds

Figure 3 demonstrates how varying cadmium concentrations affect its adsorption by RPF and CuPF.

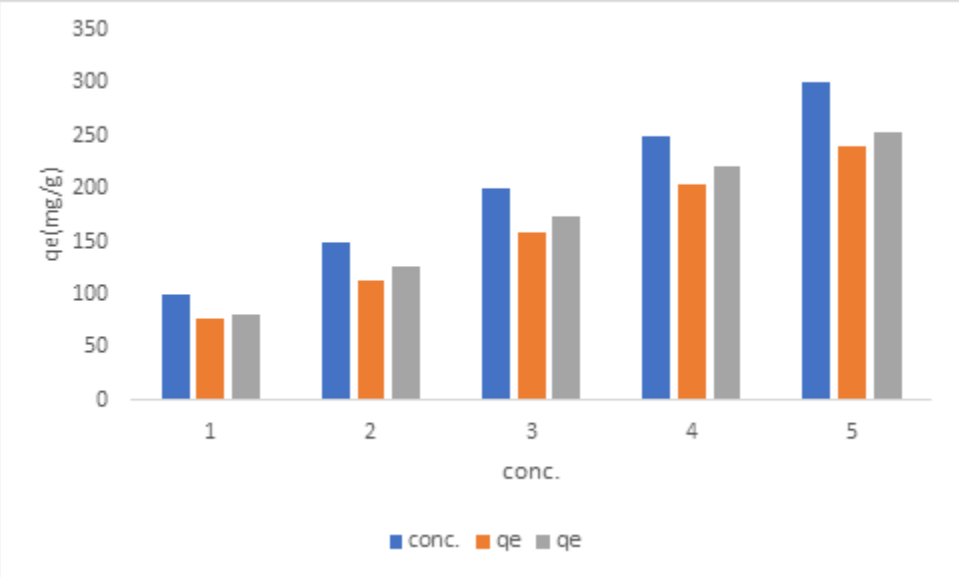

Figure 3: Plots for the effects of concentration on the adsorption of cadmium onto palm fronds.

It was seen that as the initial cadmium ion concentration rose from 100mg/L to 300mg/L, the adsorption capacity increased. This is probably due to a higher number of metal ions in the solution, which cause more frequent collisions and improved adsorption. Concentration effect was further confirmed with a one-way ANOVA test which showed no significant difference in the mean efficiency of RPF and CuPF. This means that the efficiency of raw and nano composites of adsorbents is relatively the same.

Effects of dosage on the Adsorption of cadmium onto palm fronds

The result illustrating how adsorbent dosage affects cadmium adsorption onto RPF and CuPF is shown in Figure 4a & 4b.

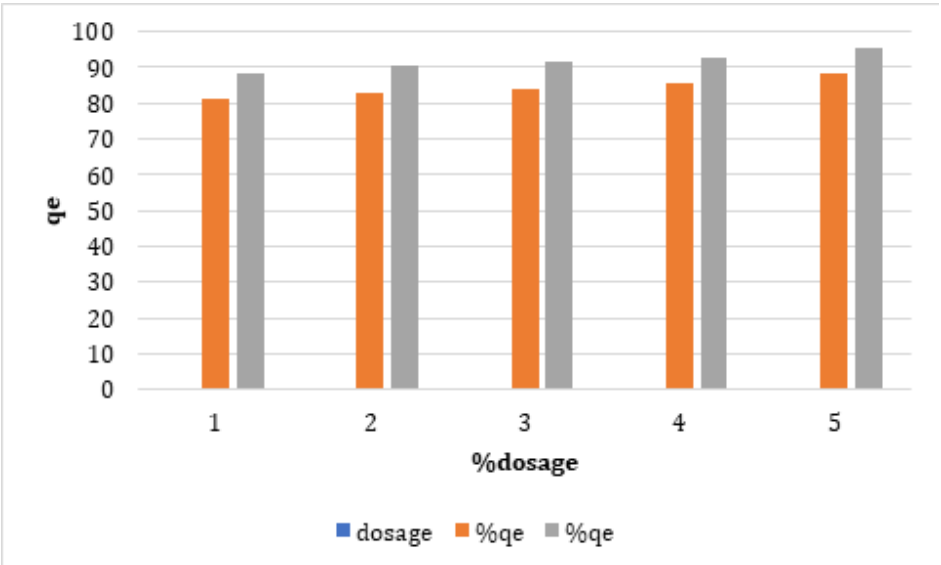

Figure 4a: Plots for the effects of percentage dosage on the adsorption of cadmium onto palm fronds.

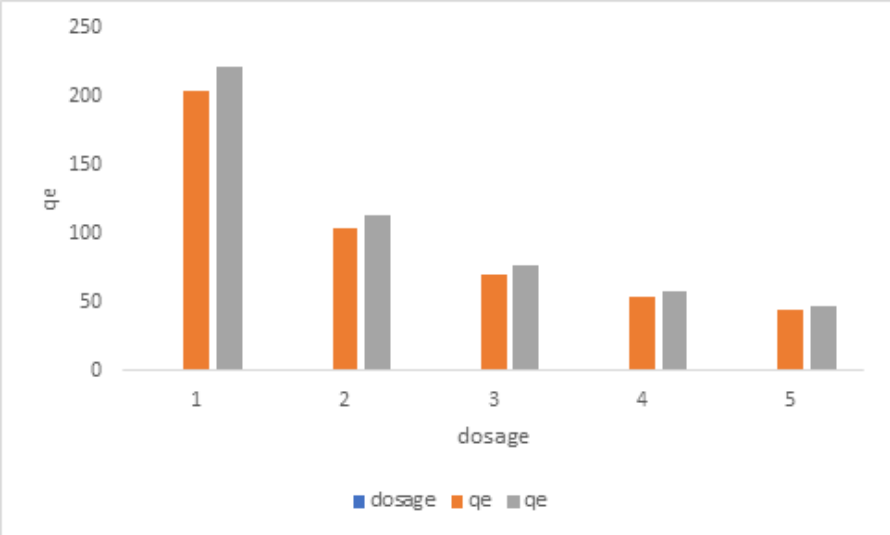

Figure 4b: Plots for the effects of dosage on the adsorption of cadmium onto palm fronds.

Figure 4a showed that cadmium removal efficiency improved from 81.4066% to 88.6046% for RPF and from 88.2692% to 95.2317% for CuPF as the adsorbent dosage increased from 0.02g to 0.1g. This enhancement is likely due to the expanded surface area and greater availability of active adsorption sites [30,31]. However, Figure 4b showed that adsorption capacity decreases with a higher adsorbent dose. This reduction is probably caused by clustering of the adsorption sites, declining the surface area available for cadmium ion attachment [32]. Although the overall cadmium removal rises with increasing adsorbent doses, the amount of cadmium adsorbed per unit mass of adsorbent declines, leading to a drop in adsorption capacity. ANOVA test showed no significant difference in the mean efficiency of RPF and CuPF. This means that the efficiency of raw and nano composites of adsorbents is relatively the same.

Effects of temperature on the adsorption of cadmium onto palm fronds

Temperature effect on the adsorption of cadmium onto RPF and CuPF was investigated and the result shown in Figure 5.

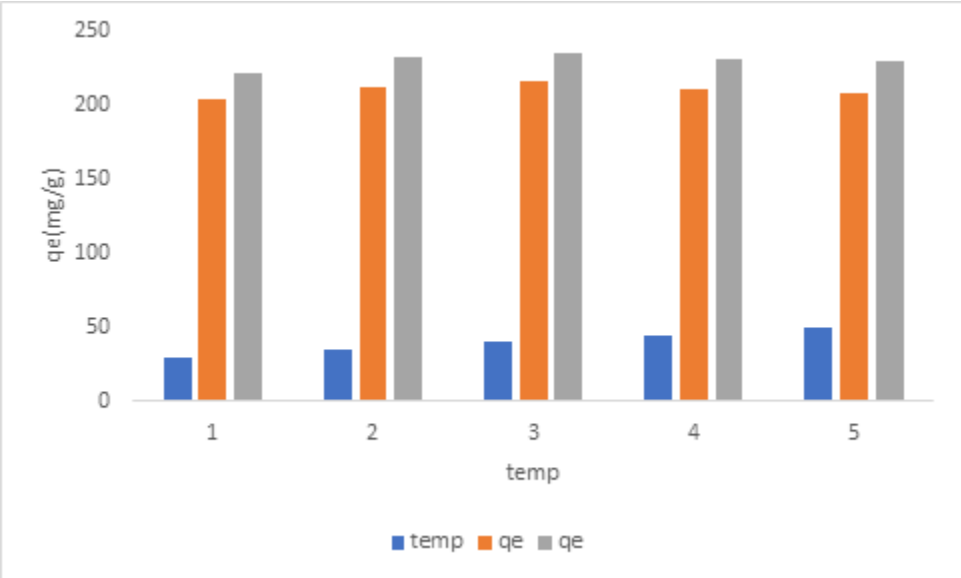

Figure 5: Plots for the effects of temperature on the adsorption of cadmium onto palm fronds.

From the result, the rise in temperature from 303K to 327K significantly enhanced cadmium removal, increasing adsorption capacities from 203.5166mg/g to 207.9002mg/g for RPF and from 220.6729mg/g to 229.4953mg/g for CuPF. ANOVA test was conducted for its statistical significance and the result showed a statistically significant difference in the mean efficiency of RPF and CuPF. It was observed that CuPF is more efficient than that of RPF (Table 2).

Table 2: ANOVA Results for all Effects on cadmium adsorption onto palm fronds.

	Sum of Squares	Df	Mean Square	F	Sig.	
Effects of time	Between Groups	594.551	1	594.551	11.083	.008
	Within Groups	536.459	10	53.646		
	Total	1131.009	11			
Effects of dosage	Between Groups	162.413	1	162.413	.036	.855
	Within Groups	36493.444	8	4561.680		
	Total	36655.857	9			
Effects of concentration	Between Groups	402.350	1	402.350	.087	.775
	Within Groups	36824.183	8	4603.023		
	Total	37226.533	9			
Effects of pH	Between Groups	615.083	1	615.083	2.810	.132
	Within Groups	1751.217	8	218.902		
	Total	2366.299	9			
Effects of temperature	Between Groups	923.919	1	923.919	37.324	.000
	Within Groups	198.030	8	24.754		
	Total	1121.949	9			

Kinetic studies

Results of kinetics of cadmium adsorption onto palm fronds

Pseudo-first and second order kinetic and intraparticle diffusion models were used to analyze the data obtained from adsorption of cadmium ion onto RPF and CuPF according to [33]. A summary of the kinetic parameters generated from these models is provided in Table 3.

According to Table 3 above, the pseudo-second order model has higher coefficient of determination (R^2) value than the pseudo-first order model for RPF and CuPF respectively. Also, the calculated adsorption capacity (qe) from the pseudo-second order model (232.5581mg/g for RPF and 238.0952mg/g for CuPF) is closer to the experimental values (224.9767mg/g for RPF and 234.7389mg/g for CuPF). The results showed that pseudo-second order model best explained the cadmium adsorption on RPF and CuPF, proving that chemisorption is the dominant mechanism. In addition, the appearance of intercepts from intraparticle diffusion plots showed that cadmium adsorption not only a surface reaction. This observation is similar to a finding elsewhere [16,18,19].

Table 3: Kinetic and intraparticle diffusion models for Cd adsorption on RPF and CuPF.

Components	RPF	CuPF
qe(experimental)	224.9767	234.7389
Pseudo first order (qe)	43.6585	25.5950
K_1	-0.0154	-0.0147
R^2	0.8869	0.9152
Pseudo second order(qe)	232.5581	238.0952
K_2	0.0006	0.0012
R^2	0.9988	0.9997
Intraparticle diffusion		
Kd	2.9010	1.7874
C	185.1600	210.2500
R^2	0.9277	0.9677

Thermodynamics studies

Results of thermodynamics for cadmium adsorption onto palm fronds

Thermodynamic parameters (ΔGº, ΔHº and ΔSº) for the sorption process were monitored to determine the heat changes, spontaneity, feasibility and disorderliness related to sorption process and their calculated values for the sorption of cadmium onto RPF and CuPF is summarized in Table 4 below.

Table 4: Thermodynamics for cadmium adsorption onto palm fronds.

Parameters	RPF	CuPF
ΔSº	3.9233	17.8069
ΔHº	0.03159	0.12138
R^2	0.0452	0.204
ΔG_0 (J/mol)303	-3719.765	-5083.8804
308	-4423.886	-6485.0031
313	-4829.8353	-7057.3887
318	-4435.5905	-6526.3486
323	-4288.6189	-6485.8312

Both RPF and CuPF recorded positive values of ΔHº and ΔSº, suggesting that the removal of cadmium ions is endothermic with high disorderliness at the metal ion/sorbent interface, corresponding with the observed increase in cadmium adsorption as temperature increased. Corresponding with the observed increase in cadmium adsorption as temperature rises. Negative ΔGº values confirmed that the sorption process is spontaneous [19].

Equilibrium studies

The results of equilibrium studies for adsorption of cadmium onto palm fronds

Isotherm modelling parameters for cadmium adsorption onto RPF and CuPF were computed following [33] and the results are presented in Table 5.

Table 5: Equilibrium Isotherm for cadmium adsorption onto RPF and CuPF.

Freundlich	RPF	CuPF
1/n	0.3529	0.6241
K_f	370.5914	1.3847
R^2	0.8009	0.8259
Langmuir	**RPF**	**CuPF**
q^o	44.4444	138.8889
B	0.0164	0.0186
R_L	0.1961	0.1769
R^2	0.7603	0.5055

The Freundlich isotherm explains heterogenous surface with sites that have different affinities for the adsorbate, leading to cooperative adsorption rather than a single monolayer adsorption used for Langmuir isotherm. From the plots of Langmuir and Freundlich models used in evaluating the isotherm for cadmium adsorption onto RPF and CuPF, it was observed that Freundlich model produced the best fit with R2 values (0.8009 and 0.8259) for both RPF and CuPF respectively. This suggested that the adsorption of cadmium ions onto RPF and CuPF occurred by multilayer coverage. The adsorption intensity is reflected by Freundlich constant (1/n) values (0.3529 and 0.6241) for both RPF and CuPF respectively fall within the favorable range of 1 to 10, suggesting the adsorption process is favorable and the adsorbents have strong affinity for cadmium ions. The higher (1/n) values for CuPF imply stronger adsorption intensity and better surface heterogeneity compared to RPF [34].

Conclusion

This research highlights the critical role of palm fronds' physicochemical properties in governing their adsorption efficiency. Elevated lignin and cellulose levels classify them as stable lignocellulosic biomass, rich in functional groups that boost adsorption. These results corroborate prior studies and affirm palm fronds' promise as a sustainable adsorbent for pollutant remediation. Rather than environmental dumping, valorizing such wastes via adsorption promotes effective waste management and ecological protection.

Authors Contributions

Conceptualization: Ikeh Obianuju Adaobi and Ojiako Eugenia Nnonye, Methodology: Nwadiogbu Joseph Onyebuchi, Validation: Ejidike Lynda, Chinyere, Review and Editing: Okonkwo Ngozi. Anastasia and Nwankwo Njideka. Veronica

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