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Green Synthesis and Characterization of Copper Nano Composites of Palm fronds for the Removal of Pb(II) ion from Aqueous Solution

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ABSTRACT

Removal of harmful heavy metals from the environment is a priority amongst scientists globally. Sorption technique using agro-waste materials as adsorbents is considered an effective and eco-friendly procedure for environmental remediation. Therefore, Raw (RPF) and copper nano composite (CuPF) of palm fronds were used as adsorbents to remove lead (II) ion from aqueous solution. The effects of contact time, pH, dosage, temperature, and concentration on the adsorbents were investigated during the adsorption process. Atomic absorption spectrometry (AAS), scanning electron microscope (SEM), Fourier transform infrared spectrometry (FTIR), and X-ray diffractometry (XRD) were used to qualify and quantify the absorbate and the adsorbents. According to the results, the materials' nature and shapes were crystalline and non-spherical, rough and waxy, as confirmed by XRD and SEM analysis. Fourier transformed infra- red spectrometry displayed the presence of OH, S-H, C=O functional groups. The results showed that in all conditions studied, that CuPF had higher adsorption capacities than the raw counterpart. Statistical analysis with a one-way ANOVA test showed that there is a significant difference in the mean efficiency of RPF and CuPF for all the conditions studied. Kinetic studies proved that the pseudo-second order model best fitted the data. The intercepts of the intraparticle diffusion models (162.48 and 180.6) for RPF and CuPF respectively, indicated that the sorption of lead onto RPF and CuPF is not only a surface reaction. Equilibrium studies showed that Langmuir isotherm model displayed the best fit for the adsorption data. The positive values of ΔH^{o} and ΔS^{o} indicate an endothermic process and an increase in the degree of disorderliness at the metal ions interface leading to an enhanced interaction and, in turn, higher lead ion adsorption. The negative values of ΔG^{o} indicates the spontaneity of the process. Conclusively, palm fronds and their nano-modified counterpart could serve as a good alternative to conventional adsorbents used to treat industrial waste water effluent due to their availability, cheapness, efficacy, and eco- friendliness.

Keywords: *Green synthesis, characterization, palm fronds, Pb-removal, equilibrium studies, kinetics, thermodynamics.*

Introduction

Environmental pollution by heavy metals and organics have been a threat to the environment and of public concern [1]. Overwhelming industrialization and civilization have tremendously increased the concentrations of these pollutants, and their rate of migration and transfer in the environment is alarming [2]. These pollutants are present in both aquatic and terrestrial ecosystems as well as in the atmosphere [3]. Humans are indirectly exposed to heavy metals through the use of water and consumption of aquatic biotas [4].

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Heavy metals and organic effluents have harmful impact on the environment, including flora and fauna [5][6]. Lead, cadmium and mercury are some of the toxic heavy metals found in the environment. They have no known biological role in living organisms. Their presence in any concentration is absolutely dangerous to health [7]. Chronic exposure to cadmium and lead can cause cancer and other severe health disorders that may culminate in mortality [8]. Therefore, they should be removed from industrial effluents before being discharged into the environment [9]. Consequently, removing these elements and other environmental pollutants has become a major challenge and a high priority amongst scientists globally [10]. Several studies revealed that many techniques have been used for heavy metal removal from aqueous solution, such as sorption , membrane process, precipitation, filtration, electro coagulation, ozonation, flocculation, biological treatment, floatation, solvent extraction, oxidation, ion exchange, ultraviolent irradiation, and photo-degradation [11][9][12][13]. Amongst the various remediation techniques, sorption which involves both adsorption and absorption, is an effective procedure for environmental remediation [14] due to its low cost, ease of operation, high efficiency, design simplicity, and re-usability [15], also being environmentally friendly [16]. Additionally, natural organic sorbent materials such as rice straw, corn cobs, peat moss, wood, cotton, milkweed floss, kapok, kenaf and wool fibers can be completely degraded in nature by biological, physical, chemical and photochemical processes and found to be favorable in aqueous solution due to their hydrophilic nature [17].

To the best of our knowledge, impregnation of copper nano composite on palm fronds for removal of Pb (II) ion from aqueous solution had not been performed by any researcher. Hence, this present study aimed to synthesize, characterize and elaborate the kinetics and thermodynamics of the adsorptions of Pb(II) ion using both raw palm fronds (PF) and copper nano composite of palm fronds (Cu PF).

Materials and Method

Fig 1: Sample of palm fronds

Samples collection

Palm fronds were collected from a bush at Adazi-ani in Anaocha Local Government Area of Anambra state, Nigeria.

Sample preparation

The material (palm fronds) was made clean by washing thoroughly under running water. The washed materials were dried properly in sunlight for (5 h for three days) and then left to dry at 65 °C in the oven. The size was reduced and sieved through No 20 and 25 British Standard Sieve (BSS Sieves) and cork in clean plastic bottle prior to all analysis. Reagents were analytical grade and were used without further purification at Graceland Laboratories, Igwebuike Street, Awka, Anambra State, Nigeria [18].

Synthesis of Copper Nanoparticles

Exactly 10.0 g of $CuCl_2.2H_2O$ was dissolved in 300 mL water in a round bottomed flask with continuous stirring for 20 min by a magnetic stirrer to get 0.2M. The stirring continued till transparent solution of 0.2M $CuCl_2.2H_2O$ is formed. About I mL of CH_3COOH was added to the solution and stirred for another 30 min. Simultaneously, 4.8g (8M) NaOH was dissolved in 15mL water in a separate beaker. The 8M NaOH was added to the previous solution dropwise under vigorous stirring with help of micropipette and stirred for another 2 h at 60^0C maintaining pH of 11. The later solution was centrifuged at 5000 rpm for 30 min. Thereafter, the precipitate was filtered by Whatman paper and washed multiple times with methanol and distilled water to remove the unreacted particles reactants and organic impurities.

The obtained particles were dried in a hot air oven at 200 ^{0}C for 20 min [19].

Nanoparticle Impregnated Biomass

The as-synthesized copper nanoparticles were dissolved in distilled water with continuous stirring using a magnetic stirrer. About 8.0 g of pulverized biomass treated with 0.1M HNO_3 was added to the solution with vigorous stirring using magnetic stirrer for 8 h, after which it was allowed to settle for 30 min, followed by centrifugation at 8000 rpm for 2 h. The obtained hybrid was filtered and dried in an oven at 70 ^{0}C for 24 h. Thereafter, was pulverized thoroughly into fine powder by the use of mortar and pestle. Afterwards, it was sieved through 100 micro meter mesh [19].

Characterization of the adsorbents

The prepared adsorbents (RPF and CuPF hybrid) were characterized to determine their functional groups, morphology and crystallinity. The functional groups of the materials were determined using FT-IR spectrometer Varian 660 MidIR Dual MCT/DTGS bundle with attenuated total reflectance) with a detector at 4 cm^{-1} resolution and 200 scans per sample. Morphology of the adsorbents were discovered with using Scanning electron microscope (SEM) of model Hitachi SU 3500 scanning microscope, Tokyo, Japan while the crystallinity test was done using Shimadu XDS 2400H diffractometer model with Cu anode control, 40kv, 30mA optics.

Adsorption Experiment

A 1000 mg/L of the lead stock solution was prepared from $Pb(NO_3)_2$ in a 1L volumetric flask. Solutions of lower concentrations 100-300 mg/L were prepared by dilution. Batch adsorption technique was utilized to evaluate the adsorbents removal of the metal ion by studying the influence of varying pH (2-10), concentrations (100-300 mg/L), bio-sorbent material dosage (0.02-0.1 g), temperature(303-323K) and contact time (30-180 min). Firstly, the pH was adjusted using 0.1M solution of either HCl or NaOH as required. The varying pH experiment was conducted by adding 0.02 g of the prepared bio-sorbent materials to 20 mL of the simulated metal ion solution, which was placed in an ultrasonic 2.5 L water filled bath and sonicated at 303 K for 30 min. At the end of the sonication time, the mixture was centrifuged at 8000 rpm for 25 min and the filtrate analyzed for lead ion concentration, using the 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 where qe(mg/g) = equilibrium adsorption capacity, representing the amount of adsorbate per unit mass of adsorbent. Co and Ce (mg/L) = initial and equilibrium concentrations of the adsorbate in the solution, V(L) = volume of the solution, m(L) = mass of the adsorbent [12].

RESULTS AND DISCUSSION

FTIR Results of palm fronds

The results of FTIR analysis of RPF and CuPF are shown in table 1 while the spectrum is displayed in figures 2 and 3 below.

Table 1: The results of FTIR analysis of RPF and CuPF

Band position for RPF (cm^{-1})	Band position for CuPF(cm^{-1})	Proposed functional groups
3298	3298	OH stretching
-	2583	S-H stretching
1820	1851	C=O stretching
1650	1577	C=O bending
1310	1317	S=O stretching
1176	1108	C-O stretching
974	974	C=C bending
601	775	C-X stretching

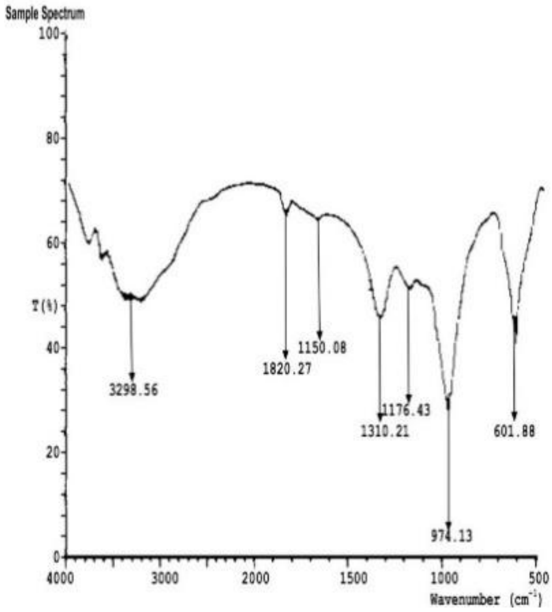

Fig.2: FTIR spectrum for RPF

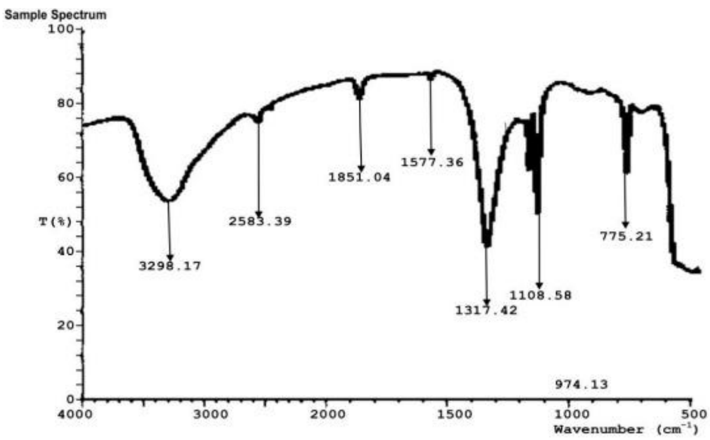

Fig 3: FTIR spectrum for Cu PF

It was observed that RPF contained OH stretching, C=O stretching, C-O stretching and C-X stretching functional groups. After modification, the FTIR spectrum of CuPF exhibited a significant difference. The presence of new bands at 2583 cm^{-1}, position of absorption linked to S-H stretching functional group, showed that incorporation of CuONPs duly modified the nature of the raw adsorbent [12][20].

SEM Results of palm fronds

The morphological properties of RPF and CuPF were determined, indicating substantial changes arising from the chemical modification process. SEM micrographs of RPF and CuPF are presented in Figures 4 and 5, respectively.

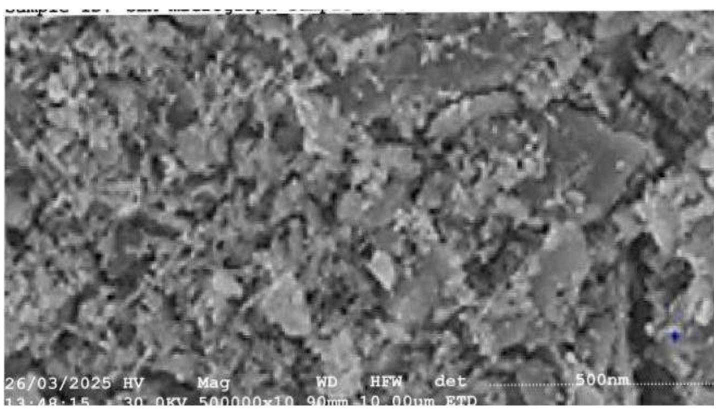

Fig 4: micrograph for RPF

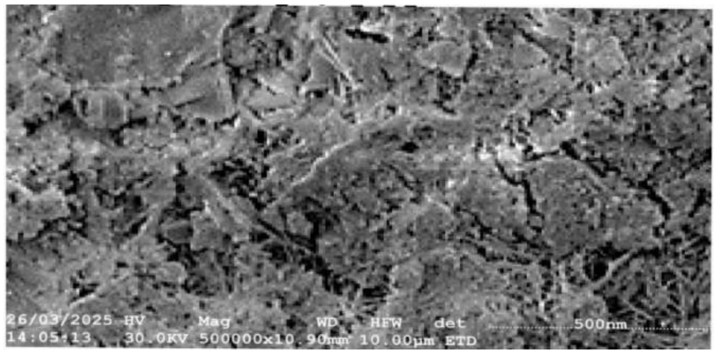

Fig 5: micrograph for CuPF

The SEM result showed that the surface of RPF was not homogeneous, had minimal irregular waxy roughness and porosity. This could limit its adsorption due to reduced surface area. After modification, the CuPF showed the presence of more evenly distributed porous and waxy- like fibers protruding across all the entire surface of the material due to the copper nanoparticles distribution on the hybrid sorbent surface. Such structural modifications expand the surface area and improve access to active sites, facilitating interactions with metal ions. Similar surface transformations following impregnation of nanoparticles have been documented elsewhere [12][13].

XRD Results of palm fronds

The XRD spectrum of RPF and CuPF are presented in figure 6 and 7 respectively.

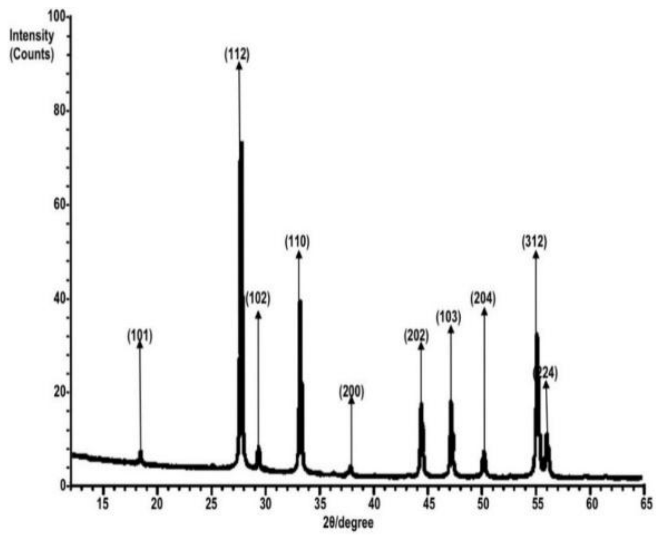

Fig 6: XRD spectrum for RPF

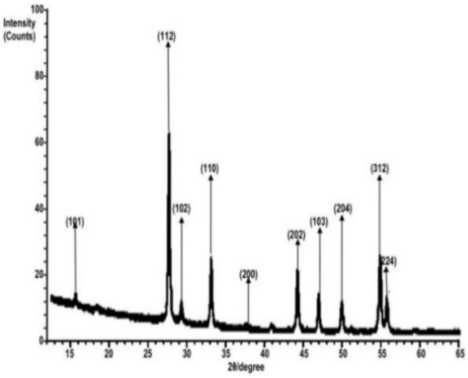

Fig 7: XRD spectrum for CuPF

The result showed that cellulose diffraction at 2Ө showed reflection at 101,102, 103,112,110,200,202, 204,224 and 312 are characteristics of typical lignocellulosic biomass with a crystalline cellulosic structure [21].

Effects of Process Parameters on the Adsorption of Lead onto palm fronds

1. Effects of Time on the Adsorption of Lead onto palm fronds

The results of the effects of time on the adsorption of lead onto raw palm fronds (RPF) and Cu nano-composites of palm fronds (CuPF) is illustrated in Figure 8.

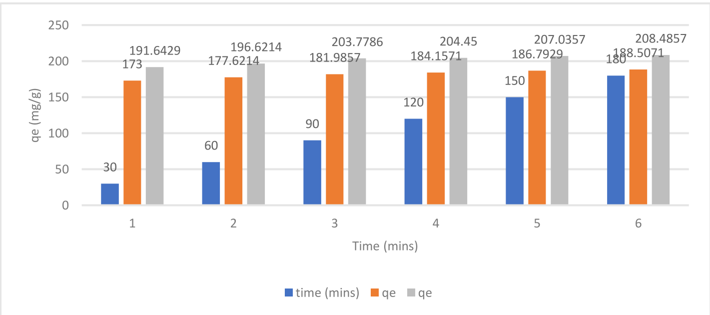

Fig 8: Plots for the effects of time on the adsorption of lead onto palm fronds

It was observed that the maximum adsorption capacity (qe) increased as the adsorption time increased from 30 to 180 minutes. The maximum adsorption capacity (qe) was minimum at 30 min and recorded to be (173.0000 mg/g and 191.6429 mg/g) for RPF and Cu PF respectively, while the maximum qe for RPF (188.5071 mg/g) and CuPF(208.4857 mg/g)occurred at 180 min. The rapid adsorption at first instance could be attributed to adherence of the adsorbate onto the surface of the materials and subsequent penetration into the available spaces [22][20][23]. The gradual decrease in adsorption rate over time could be caused by a process controlled by attachment, resulting from a reduced number of available active adsorption sites [14]. Throughout all the adsorption durations examined, CuPF exhibited a greater 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 [12][17]. The effect of time on the adsorption of lead on palm fronds was done with a one-way ANOVA test for its statistical significance, and the results are presented in Table 2. 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.

Effect of pH on the Adsorption of lead onto palm fronds

The effect of solution pH on the adsorption of lead (Pb) onto RPF and CuPF was demonstrated as shown in Fig 9.

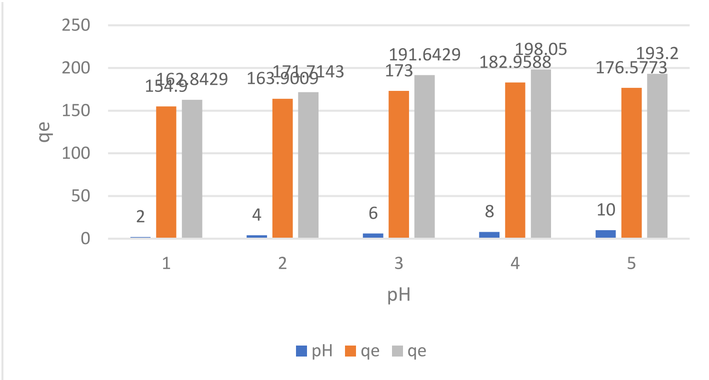

Fig 9: Plots for the effects of pH on the adsorption of lead onto palm fronds

The result indicated that increasing the pH leads to an increase in the maximum adsorption capacity. At pH 2, the adsorption capacities were relatively low (154.90000 and 162.8429 mg/g for RPF and CuPF, respectively), reaching peak values at pH 8 (182.9588 and 198.0500 mg/g) for both RPF and CuPF. However, at pH 10, a notable decrease in adsorption capacity was observed (176.5773 and 193.2000 mg/g) for RPF and CuPF, respectively, likely due to repulsive effects stemming from a reduction in available active binding sites. A one-way ANOVA was used to test for its statistical significance, and the result is presented in Table 2. It was observed that no significant difference in the mean efficiency of the adsorbents. Therefore, the efficiency of RPF and CuPF is relatively the same.

Effects of concentration on the adsorption of lead onto palm fronds

Demonstration of how varying lead concentrations affect its adsorption by RPF and CuPF is shown in Figure 10.

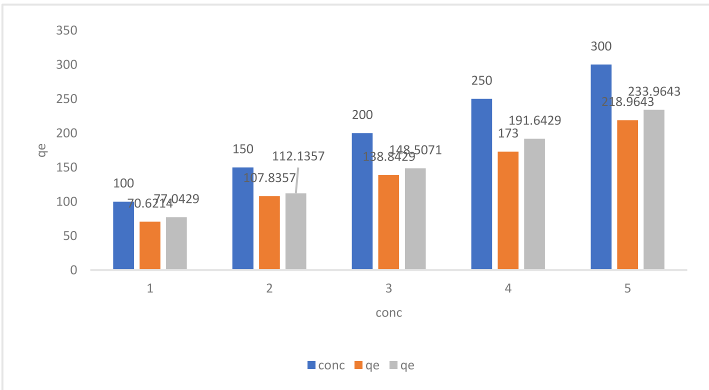

Fig 10: Plots for the effects of concentration on the adsorption of lead onto palm fronds

The result showed that when the initial concentration of lead ions increased from 100 mg/L to 300 mg/L, the adsorption capacity also increased. This is likely because a greater number of metal ions in the solution led to more frequent collisions, enhancing adsorption efficiency. To understand the directions of the effects of concentration on the adsorption of lead on palm fronds, a one-way ANOVA was used to test for its statistical significance, and the result is presented in Table 2. There is no significant difference in the mean efficiency of the adsorbents. Therefore, the efficiency of RPF and CuPF is relatively the same.

Effects of dosage on the Adsorption of lead onto palm fronds

The result illustrating how adsorbent dosage affects lead adsorption onto RPF and CuPF is shown in Fig 11a and 11b.

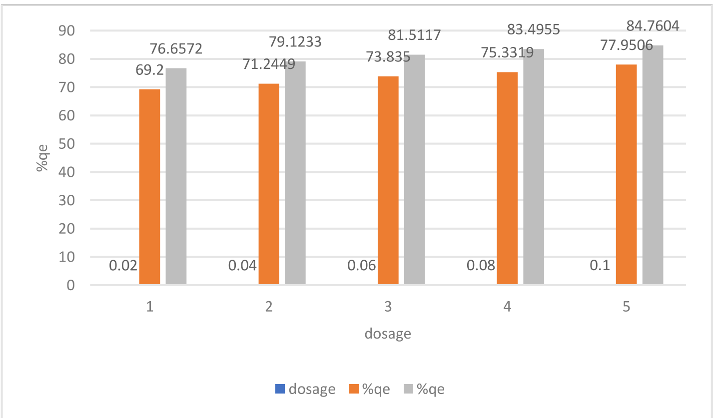

Fig 11a: Plots for the effects of percentage dosage on the adsorption of lead onto palm fronds

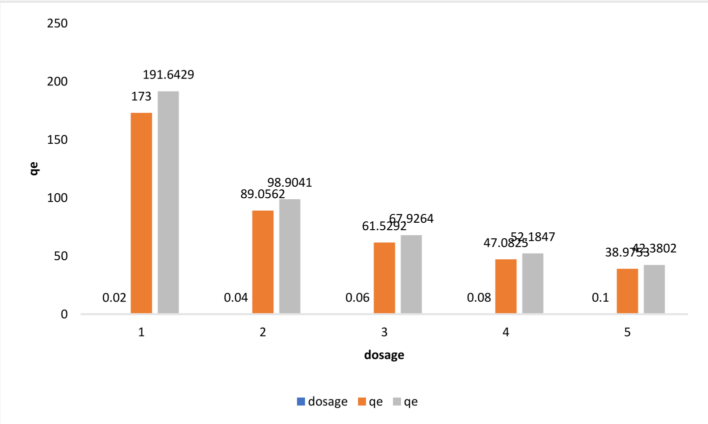

Fig 11b: Plots for the effects of dosage on the adsorption of lead onto palm fronds

From the result, increasing the adsorbent dose from 0.02 g to 0.1 g led to a rise in lead removal efficiency, ranging from 69.2000% to 77.9506% for RBH and 76.6572% to 84.7604% for CuBH. This improvement is likely due to a larger surface area and more active sites available for adsorption [24]. Conversely, Fig. 11b displays a decline in adsorption capacity as the adsorbent dose increases. This decrease could be attributed to the overlapping of adsorption sites, which reduces the total available surface area for lead ion binding [22]. Hence, while the total lead removal increases with more adsorbent, the amount adsorbed per unit weight of adsorbent drops, lowering the adsorption capacity [25]. A one-way ANOVA was used to test for its statistical significance, and the result is presented in Table 2. The result showed no significant difference in the mean efficiency of the adsorbents. Therefore, the efficiency of RPF and CuPF are relatively the same.

Effects of temperature on the adsorption of lead onto palm fronds

In this study, the influence of temperature on the adsorption of lead onto RPF and CuPF was investigated, with the results shown in figure 12.

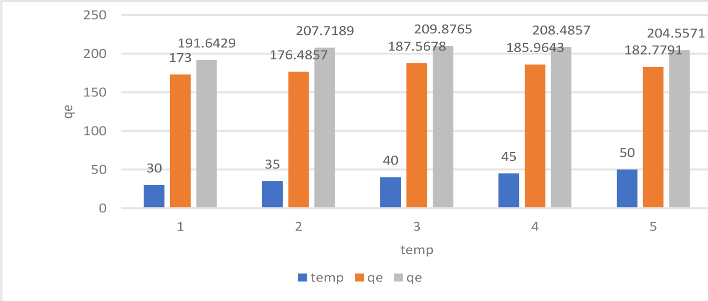

Fig 12: Plots for the effects of temperature on the adsorption of lead onto palm fronds

Temperature plays a crucial role in the removal of pollutants from solutions. It was found that increasing the temperature from 303 K to 313 K led to a significant rise in lead removal, with adsorption capacities increasing from 173.0000 mg/g to 187.5678 mg/g for RPF and from 191.6429 mg/g to 209.8765 mg/g for CuPF. However, a decline in adsorption efficiency was observed as the temperature further increased from 318 K to 323 K, with (qe) values decreasing from 185.9643 mg/g to 182.7791 mg/g for RBH and 208.4857 mg/g to 204.5571 mg/g for CuPF, respectively. The decrease in adsorption could be due to increased kinetic energy of the adsorbate molecules at higher temperatures, which can lead to easier desorption and weaker interaction forces between the adsorbate and the adsorbent surface, thereby reducing the adsorption capacity. A one-way ANOVA was used to test for its statistical significance, and the result is presented in Table 2. There was 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: ANOVA Results for all Effects on lead adsorption onto palm fronds

ANOVA						
		Sum of Squares	Df	Mean Square	F	Sig.
Effects of time	Between Groups	1199.002	1	1199.002	31.312	.000
	Within Groups	382.926	10	38.293		
	Total	1581.929	11			
Effects of dosage	Between Groups	188.313	1	188.313	.057	.817
	Within Groups	26413.777	8	3301.722		
	Total	26602.090	9			
Effects of concentration	Between Groups	291.909	1	291.909	.081	.783
	Within Groups	28663.933	8	3582.992		
	Total	28955.842	9			
Effects of pH	Between Groups	437.094	1	437.094	2.458	.156
	Within Groups	1422.760	8	177.845		
	Total	1859.854	9			
Effects of temperature	Between Groups	1356.857	1	1356.857	28.896	.001
	Within Groups	375.650	8	46.956		
	Total	1732.507	9			

Kinetic Studies

Kinetics of lead adsorption onto palm fronds

The adsorption of lead onto RPF and CuPF was analyzed using experimental data fitted to the pseudo-first order, pseudo-second order and intraparticle diffusion kinetic models. A summary of the kinetic parameters obtained from these models is provided in Table 3.

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

Components	RPF	CuPF
qe(experimental)	188.5071	208.4857
Pseudo first order (qe)	29.9371	33.7034
K_1	-0.0177	-0.0199
R^2	0.9648	0.9573
Pseudo second order(qe)	192.3076	212.7659
K_2	0.00116	0.00115
R^2	0.9998	0.9999
Intraparticle diffusion		
Kd	1.9754	2.1650
C	162.4800	180.6000
R^2	0.9945	0.9551

From Table 3, the coefficient of determination (R^2) value for the pseudo-second order model (0.9998 for RPF and 0.9999 for CuPF) is higher than that for the pseudo-first order model (0.9648 for RPF and 0.9573 for CuPF). Additionally, the calculated adsorption capacity (qe) from the pseudo-second order model is closer to the experimental value. The results indicate that the pseudo-second order model more accurately described the lead adsorption on RPF and CuPF, suggesting that chemisorption is the dominant mechanism. Also, the intercept from the intraparticle diffusion plots (162.48 for RPF and 180.6 for CuPF) indicates that lead sorption involves both surface reaction and diffusion into the adsorbent's inner pores. These findings align with studies in the literature [26][12][17].

Thermodynamics studies

Thermodynamics for lead adsorption onto palm fronds.

The sorption thermodynamic parameters (ΔGº, ΔHº, and ΔSº) provide valuable insights into the heat changes, spontaneity, feasibility, and disorderliness associated with the sorption process, and their calculated values for the sorption of lead onto RPF and CuPF are summarized in Table 4.

Table 4: Thermodynamics for lead adsorption onto palm fronds

Parameters	RPF	CuPF
ΔSº	21.7028	21.4052
ΔHº	0.09477	0.1089
R^2	0.5300	0.3012
ΔGº 303	-2038.9935	-2995.2598
308	-2242.4155	-4076.1413
313	-2862.5102	-4305.4755
318	-2818.6106	-4266.6483
323	-2685.9590	-4039.6803

The positive ΔHº values for both RPF and CuPF indicate that the removal of lead ions is endothermic, corresponding with the observed increase in lead adsorption as temperature rises. Positive ΔSº values suggest a rise in disorder at the metal ion/sorbent interface, which enhances interaction and leads to greater metal ion adsorption. Negative ΔGº values confirm that the sorption process is spontaneous [12].

Equilibrium studies

Equilibrium studies for adsorption of lead onto palm fronds

The derived isotherm parameters for lead adsorption onto RPF and CuPF are presented in Table 5.

Table 5: Equilibrium Isotherm for lead adsorption onto RPF and CuPF

Freundlich	RPF	CuPF
Xn	0.2319	0.2957
K_f	979698.3528	5668.0476
R^2	0.9089	0.9595
Langmuir	RPF	CuPF
qo	22.3214	35.3357
b	0.0113	0.0143
R_L	0.2614	0.2186
R^2	0.9555	0.9922

From the R^2 values, it was observed that the Langmuir model produced the best fit (0.9555 and 0.9922) for both RPF and CuPF, respectively. This implies that the adsorption of lead ions onto RPF and CuPF occurred by a monolayer coverage.

Conclusion

This research work showed that palm fronds, which are common agro-waste materials in Nigeria, can be utilized as adsorbents in the remediation of wastewater. Spectral analysis done showed structural alterations indicating the presence of new species. In all the conditions studied, the copper nano composites of the adsorbents showed a higher adsorption efficiency than the raw materials. This was confirmed by a statistical test of significant difference with a one-way ANOVA, which confirmed that the nanocomposites of the sample materials were more effective than the raw counterparts. The kinetic studies of both raw and modified Bambara nut hulls and palm fronds revealed that the sorption occurred by surface reaction and diffusion into the pores of the materials. The Langmuir isotherm model best fitted the data. The thermodynamic studies indicated that the reactions were endothermic with an increased degree of disorderliness and spontaneity of the process. Therefore, the speed of removal and high adsorption capacity of the copper nanocomposite materials make it a promising alternative adsorbent for heavy metal adsorption, considering that it is cheap, biodegradable, and readily available.

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