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Characterization and Adsorption Capacity Evaluation of $ZnCl_2$ Impregnated Cassava Peel Carbon for Removal of Fe, Ca, Mg and Zn ions in Wastewater from Cassava processing Industry

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ABSTRACT: The objective of this paper was to prepare, characterize and evaluate the adsorption capacity of cassava peel carbon (CPC) impregnation with $ZnCl_2$ salt for the removal of Fe, Ca, Mg and Zn ions in wastewater derived from cassava processing which has been noted to be problematic in terms of management. CPC and cassava peel activated carbon (CPAC) at 1:3, 2:3, and 1:1 impregnation levels were placed in fixed bed adsorption columns to determine metal ion adsorption capacities of the carbon materials after 120, 240, 360 and 480-minute contact times. Langmuir and Freundlich isotherm models were used in assessment of adsorption of the carbon materials, while pseudo-first and second order models were used to validate the adsorption kinetics of Fe, Ca, Mg and Zn. Pore space development was also monitored using scanning electron microscope (SEM) imagery. Results SEM images revealed hexagonal honeycomb surface structure at 2:3 impregnation level and spongy configuration at 1:1 $ZnCl_2$ impregnation level. The correlation coefficient of Langmuir isotherm was observed to be between 0.86 to 1, indicating that the adsorbent is very good in adsorption of the metals, however, Freundlich isotherm is found to be unfavorable in describing CPAC's capacity in adsorbing zinc ion contaminant. The pH of the carbon materials was observed to have increased while the bulk density reduced with increasing level of impregnation. Conclusively, CPC and CPAC materials are well suited to Langmuir isotherm as well as pseudo-second order model, implying that adsorption occurs well in a monolayer form on the surface material surface.

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Metal ion contamination of water bodies is a serious environmental concern that endangers ecosystems and human health. Water bodies can be exposed to metal ions from a variety of sources, such as inappropriate waste disposal, industrial discharges, agricultural runoff and so on (Zhang *et al*. 2023). These metal ions can build up in sediments and aquatic life when they get into the water, and eventually they can make their way up the food chain (Singh *et al*. 2022). According to Das *et al.* (2023) Elevated levels of metal ions have the potential to upset ecosystem balances, resulting in decreased biodiversity and population decreases. It has also been observed by Jaishankar *et al.* (2014) that for aquatic life, some metal ions can be hazardous resulting in reproductive issues, organ damage, and even death. Furthermore, individuals who consume contaminated seafood stand risk of contracting diseases caused by metal ions that bioaccumulate in the tissues of fish and other aquatic creatures (Khushbu, 2022). The health of humans is negatively impacted by metal ion contamination of water bodies in addition to its environmental consequences. Drinking water tainted with excessive concentrations of metal ions can cause renal damage, neurological

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difficulties, and developmental problems in children, amongst other medical conditions. Children and pregnant women are among the vulnerable groups particularly at risk from drinking water contaminated with metal ions. Cassava peels are a byproduct of cassava processing, research has shown that they can be processed to give activated carbon, which is an effective material for adsorbing contaminants in water as observed by Omotosho and Sangodoyin (2013). The application of activated carbon derived from cassava peels in water treatment has garnered interest as an economical and environmentally friendly method of eliminating impurities from water sources. Studies have also shown that cassava peel activated carbon has very good adsorption characteristics when it comes to metals and heavy metals from water. (Adeolu and Adewoye 2019; Rajeshwarisivaraj *et al.*, 2001; Alongamo *et al.*, 2021).

By definition, adsorption is the process by which molecules or ions adhere to the surface of a solid material, like carbon, while kinetics is the rate at which a substance is adsorbed onto a medium. Studies have shown that for carbon materials such as activated carbon derived from sources such as coconut shells, wood, or agricultural waste like cassava peel, the kinetics of adsorption play a significant role in determining how quickly pollutants or contaminants are removed from a solution. The surface area, pore structure, and chemical structure of the carbon all affect the rate at which molecules or ions are adsorbed onto carbon materials' surfaces. According to Lazarević-Pašti *et al*. (2023), a number of variables, such as the temperature, pH, and type of adsorbate-adsorbent interaction, can affect the kinetics of adsorption in carbon-based materials. Understanding and maximizing the effectiveness of adsorption processes however depends heavily on the link between kinetics and adsorption in carbon-based materials. In designing and operating adsorption systems for effective pollutant removal, it is therefore essential to understand the kinetics of adsorption. Hence, the objective of this paper was to prepare, characterize and evaluate the adsorption capacity of cassava peel carbon (CPC) impregnation with $ZnCl_2$ salt for the removal of Fe, Ca, Mg and Zn ions in wastewater obtained from cassava processing activities.

MATERIALS AND METHODS

Sample collection and preparation: Cassava peels used for this study were sourced from a local cassava processing factory were inspected, washed and sun dried to a moisture content of between 8-11% dry-basis. Dried cassava peels were wrapped in aluminium foil then carbonized in Gallenkamp muffle furnace at 420°C for 90 mins. It was then allowed to cool overnight under inert conditions. The produced cassava peel carbon (CPC) was taken through a size reduction process using a pestle, mortar and sieve until a size of 500 µm was achieved. The powdered CPC material was pre-washed to remove any form of dirt that may have been attached during size reduction process and later dried in the oven at 120°C for 8 hrs. The carbon material was then activated with zinc chloride salt at different carbon to salt ratios (0:1, 1:3, 2:3, 1:1). In order to ensure complete reaction of the impregnation salt ($ZnCl_2$) with the CPC, the samples were subjected to temperature range of 200-450°C for 6 hrs. The pH was then stabilized to neutral by washing each cassava peel activated carbon (CPAC) sample in slightly boiling deionized water to remove the residual zinc chloride salt. After drying in an oven at 90°C for 24 hrs, the materials were taken out and allowed to cool at ambient temperature before being stored away in glass jars until used.

Characterization of activated cassava peel carbon: The pore space development on surfaces of the CPC and CPAC samples were monitored by focusing high energy electrons from a scanning electron spectroscope (SEM, EVO/MA15) at Sheda Science and Technology complex, Gwagwalada Abuja, Nigeria with a range of signals to produce the micrographs. Pore space and surface area development as shown on these micrographs were then read with the aid of image J (an interactive image processing Java software). The water samples obtained from the adsorption experiment were analyzed according to WHO and EC standards.

Fixed adsorption bed experiment set-up: The adsorption studies were carried out using fixed adsorption bed experimental set-up. Four fixed adsorption bed adsorption columns of dimensions 14 x14 cm and height 70 cm were constructed from transparent Perspex material. Each of the fixed bed adsorption columns were then filled with 400 grams of CPC and CPAC at 1:3, 2:3 and 1:1 $ZnCl_2$ activation ratios. The cassava processing wastewater sample collected from a cassava processing factory at Odo-Ona area of Ibadan, Oyo state was placed in an elevated reservoir and allowed to flow onto the fixed bed adsorption columns. The wastewater was retained in each of the filters for a time ranging between 120 to 480 minuites before flowing down the filtering bed, the procedure was replicated five times as recommended by Ijaola *et al.*, (2013). The effluent collected from the treatment setup was subjected to chemical analysis to determine the Fe, Ca, Mg and Zn contents of the effluent using spectrophotometry using Atomic Adsorption Spectrophotometer machine with

HNO3 as the reagent. The adsorptive capacity of the CPC and CPAC were thereafter evaluated using pseudo-first and second order kinetic models while the adsorptive isotherms were done by using Langmuir and Freundlich isotherms.

Effect of concentration and time: The difference between the initial and final concentration over the contact time, which ranged from 120 to 480 minutes, was used to determine the amount of metal ions adsorbed by the carbon materials.

CPC and CPAC adsorption isotherms: In order to effectively capture the phenomenon of adsorption of ions in wastewater by adsorbents the most common isotherms used are the Freundlich and Langmuir isotherms. This is because these isotherms have been observed to adequately reflect the capacity of activated carbon materials in adsorbing pollutants (Elhussien and Isa, 2015; Obiad, 2020). This Freundlich isotherm is mathematically expressed as shown in equation 1.;

$$log q_e = log k_f + \frac{1}{n}(log C_e) \quad (1)$$

Where q_e is the equilibrium metal ion concentration on the adsorbent (mg/g dry weight), C_e is the equilibrium metal ion concentration in the solution (mg/L), K_f is a constant that describes the adsorption capacity of the adsorbent and n is an empirical parameter which dictates the intensity of the adsorption and the Langmuir adsorption isotherm is presented in equation 2.

$$\frac{C_e}{q_e} = \frac{1}{Q_m K_L} + \frac{C_e}{Q_m} \quad (2)$$

where C_e is the equilibrium metal ion concentration in the solution (mg/l), q_e is the metal ion concentration on the adsorbent at equilibrium (mg/g), qm is the monolayer adsorption capacity of the adsorbent (mg/g) and K_L is the Langmuir adsorption constant(l/mg).

Adsorption Kinetics: The adsorption kinetics theory is hinged on the interactions between the adsorbate, adsorbent and the system conditions. The Pseudo-first order rate of equation also referred to as the Lagergren model used is expressed as equation 3 (Edet and Ifelebuegu, 2020).

$$\frac{dq_t}{d_t} = k_t(q_e - q_t) \quad (3)$$

Where qe is the adsorption capacity of the adsorbent at equilibrium (mg.g^{-1}), *qt* is the adsorption capacity of the adsorbent at time *t* (mg.g^{-1}) and K_t is the rate constant for pseudo first order adsorption (min^{-1}). Therefore, applying first-order model of adsorption, the plot of log (q_e-q_t) against (t) gave a linear relationship from which values of and k_t were determined from the slopes and intercepts of the plots.

As shown by Ho and McKay (1998), the second order model is a chemisorptions kinetic rate equation which describes the rate of sorption and according to Ademiluyi and Nze (2016) it can be expressed as equation 4.

$$\frac{d_{qt}}{d_t} = K(q_e - q_t)^2 \quad (4)$$

Where d_q and d_e are the sorption capacity at equilibrium and at time t, respectively in mg/g and k, the rate constant of pseudo-second order of sorption (g/mg/min). If equation 4 is integrated and rearranged with (mg/g/min) as the initial adsorption rate, since $h_0 = kq_e{}^2$ when ${}^{d_{qt}}/_{t}$ → 0 equation 4 becomes

$$\frac{t}{q_t} = \frac{1}{h_0} + \frac{1}{q_e t} \quad (5)$$

Hence, applying second-order model of adsorption, the plot of ${}^{t}/_{q_t}$ against *t* gave a linear relationship from which values of q_e and t determined from the slopes and intercepts of the plots.

RESULTS AND DISCUSSION

Studies show that adsorbent's surface may become inactive with increase in temperatures due to loss of active portion caused by breaking of bonds, reducing the force of attraction between the adsorbent and adsorbate. The temperature of the CPC and CPAC was kept constant, according to the results displayed in Table 1, indicating that temperature had no bearing on the adsorption investigations. Studies carried out by Parvathi (2007) revealed that higher pH values cause ligands to rise negatively, thereby increasing the cation's ability to bind. Zinc chloride's capacity to attack metal oxide in carbon and produce a derivative may be a probable cause of this phenomenon. The density was also observed to have reduced with an increase in activation level of the carbon material which does not align with observations made by Akpa and Dagde (2018).

Table 1: Bulk density, pH and temperature values for variation in zinc chloride ratio of carbon derived from cassava peel

Parameters	**Activation Ratio**			
	0:1	**1:3**	**2:3**	**1:1**
pH	5.3	5.3	5.5	5.7
Bulk Density (g/m^3)	0.407	0.402	0.397	0.391
Temp (ºC)	27	27	27	27

Table 2: Progressive Adsorption of metals ions on CPC and CPAC Materials over Time (mg)

Time (mins)	Fe^{2+}				Ca^{2+}				Mg^{2+}				Zn^{2+}			
	0:1	1:3	2:3	1:1	0:1	1:3	2:3	1:1	0:1	1:3	2:3	1:1	0:1	1:3	2:3	1:1
0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
120	0.10	0.33	0.35	0.35	4.21	6.23	4.33	4.21	2.81	4.62	3.62	4.42	0.04	0.09	0.09	0.14
240	0.12	0.36	0.37	0.38	5.23	7.23	6.23	5.10	3.53	4.32	4.64	4.63	0.04	0.09	0.09	0.09
360	0.23	0.37	0.37	0.38	7.22	7.45	6.45	6.01	3.64	4.52	5.63	5.54	0.06	0.09	0.08	0.08
480	0.23	0.38	0.37	0.37	7.34	7.66	6.56	6.0	3.66	4.62	5.65	5.02	0.08	0.08	0.08	0.08

Results of progressive adsorption of metal ions in the carbon materials as shown on Table 2 revealed that the rate of metal ion removal increased with level of activation as also observed by Ijaola *et al*. (2013) as well as Omotosho and Sangodoyin (2013). This may have been as a result of availability of large surface area in CPC and pore space development resulting from $ZnCl_2$ impregnation leading to increased surface area as well as the effect of increases in negative charges on the surface thereby facilitating metal uptake within the pores of the CPAC. Results also show that progressive adsorption was of Fe was observed from the start of the experiment till about 360 mins contact time when residual ion concentration became constant indicating that the CPC material had reached an adsorption saturation point. For the CPAC it was however noticed that although a similar trend of increase in progressive adsorption level was observed for Fe ions as observed in CPC there was however noted that at 1:1 zinc chloride activation there was an onset of desorption at 480 mins contact time, this may have been due to the weakening of surface negative charges on the carbon material and the Fe metals. The effect of metal ion desorption was more pronounced for the zinc ion, it was noted that the onset time for desorption reduced with increase in activation levels of the CPAC materials, this observed trend may have been as a result of presence of residual zinc metal from the activation salt ($ZnCl_2$).

Effect of Activation on Pore Space Development: The micrograph image of CPC (Plate 1) obtained from the scanning electron microscope showed that the carbon material has relatively rough surface which is indicative of large surface area for adsorption. At $ZnCl_2$ activation ratio of 1:3 the micrograph obtained revealed that there was relatively larger pore opening characteristics when compared to the CPC as shown in Plate 2. Image from the CPAC at 2:3 Zinc Chloride activation level reveals that the carbon material developed a hexagonal honeycomb surface pore structure (Plate 3) this also theoretically indicates good adsorptive surface characteristics. Plate 4 shows that the CPAC had a spongy shape at 1:1 zinc chloride activation, suggesting that the activation process had produced pores on the carbon material's surface. This structure also demonstrates that, in theory, the material will make an excellent adsorbent.

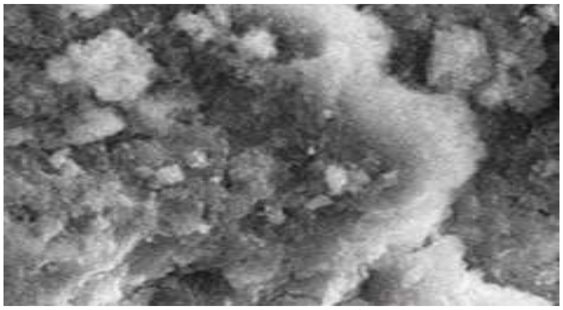

Plate 1: Micrograph of CPC

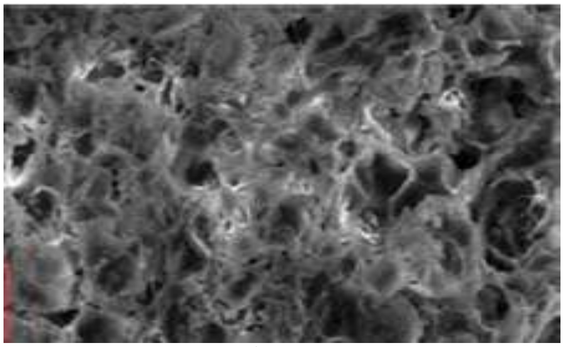

Plate 2: Micrograph of CPAC at 1:3 activation ratio

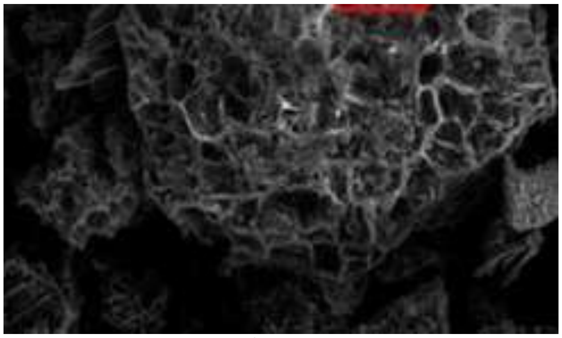

Plate 3: Micrograph of CPAC at 2:3 activation ratio

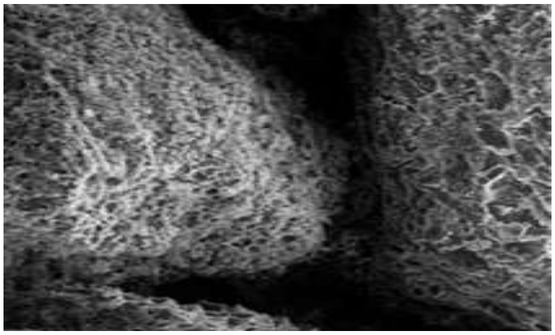

Plate 4: Micrograph of CPAC at 1:1 activation ratio

Langmuir Isotherm Fitting: According to Ambursa *et al.*, (2011), high values of Q_m and K_L indicates that adsorption it is more favourable for the ions in question; hence greater efficiency. The constants Q_m and K_L as stated in Table 3 represent the Langmuir monolayer saturation capacity and Langmuir isotherm constant

Table 3: Comparison of Langmuir model Adsorption isotherms constants at different $ZnCl_2$ activation levels

Activation ratio	Langmuir Constants	Metal ion			
		Fe^{2+}	Ca^{2+}	Mg^{2+}	Zn^{2+}
1:0	R^2	1	0.87	0.86	1
	K_L	12.93	0.34	0.52	0.60
	Q_m	1.82	25.73	18.32	11.34
	K_a	0.93	1.33	1.39	0.91
1:3	R^2	1	1	0.99	0.98
	K_L	14.21	2.34	3.22	0.33
	Q_m	12.34	2.34	1.093	12.22
	K_a	2.33	2.45	2.55	1.12
2:3	R^2	0.94	0.98	1	0.94
	K_L	12.56	5.22	3.42	0.0023
	Q_m	10.03	0.00056	0.0043	0.123
	K_a	2.56	2.68	2.53	1.13
1:1	R^2	1	0.99	0.99	0.89
	K_L	5.22	5.33	3.12	0.0012
	Q_m	0.004	0.00021	0.0032	0.044
	K_a	2.92	2.69	2.52	1.11

Freundlich Isotherm Fitting: When compared to the Langmuir Isotherm this isotherm gives an overview of multi-layer, empirical adsorption characteristics on homogeneous sites while that of Langmuir is monolayer basis and much of theoretical basis. The adsorption process is considered favorable when the value of 1/n is between 0.1 and 1.0 as observed by Awad *et al.*, (2023), it also indicates a larger change in effectiveness over different equilibrium concentrations. Also, when 1/*n* is >1.0, the change in adsorbed concentration is greater than the change in the solute concentration as observed by Chilton et al (2002). The adsorption constants K_f and1/n were obtained from the slope and intercepts of log q_e against C_e plot for each ion at different activation levels. The result (Table 4) revealed that the 1/n values for Fe and Ca ion were favourable for CPC and CPC at 1:3 and 2:3 activation levels only. For Mg the 1/n constant values as revealed by Freundlich isotherm showed a favourable adsorption condition for the CPC as well as CPAC at all levels. The Zn ion adsorption by CPC was favourable while the CPAC exhibited an unfavourable condition which increased with activation level. This inferred that Freundlich isotherm is found to be unfavourable in describing CPAC's capacity in adsorbing zinc ion contaminant in wastewater, which were obtained from the intercepts and slopes of the linear plots of Langmuir isotherms for adsorption for each of the metals (Fe, Ca, Mg and Zn) on CPC and CPAC. For Fe it was observed that there was an increase in the Q_m and K_L values at 1:3 and 2:3 $ZnCl_2$ activation levels when compared to CPC (0:1) this shows that the increase in activation level made significant impact on the monolayer adsorption of Fe, however at 1:1 activation level it was observed that there was a drop in the values of the constants suggesting that there may have been some form of desorption or reduction in availability of sites for adsorption on the activated material.

For Zn it was however observed that the Q_m and K_L values reduced with increase in activation level thereby indicating that the material had poor affinity for Zn ion adsorption, this may have been due to the effect of residual zinc salt content in the CPAC inhibiting the process. The value of the correlation coefficients R^2 is an indication of how well suited a model is for adsorption Odogu *et al.* (2020), thus a relatively high value is preferred. Results in Table 3 shows that the R^2 value of the four metals for CPC and CPAC were between 0.86 to 1, indicating that the adsorbent is very good in adsorption of these metals. Comparison of these R^2 values also revealed that zinc chloride activation improved the efficiency of Ca and Mg adsorption from the observed values of 0.87 and 0.86 respectively for CPC to a range of between 0.98 to 1 R^2 values.

Table 4: Freundlich Constants

Activation ratio	Freundlich Constants	Metal ion			
		Fe^{2+}	Ca^{2+}	Mg^{2+}	Zn^{2+}
1:0	R^2	1	1	1	1
	K_f	0.93	0.34	0.52	0.60
	1/n	0.22	0.23	0.32	0.34
1:3	R^2	1	1	1	1
	K_f	0.21	0.34	0.22	0.33
	1/n	0.34	0.34	0.49	0.09
2:3	R^2	1	1	1	1
	K_f	0.56	0.22	0.42	0.023
	1/n	0.50	0.56	0.43	0.09
1:1	R^2	1	1	1	1
	K_f	5.22	1.33	0.12	0.012
	1/n	0.09	0.021	0.32	0.044

Pseudo-first order equation Fitting: Figs. 1-4 show the plots of First order model adsorption kinetics for Fe, Ca, Mg and Zn ions removal adsorption with by CPC and CPAC impregnated at 1:3, 2:3 and 1:1 activation ratios. The plot for activated and non-activated cassava peel carbon materials revealed that the plot for adsorption of Fe by CPC were fairly straight. The plots for Mg at 2:3 and 1:1 as well as Zn at 2:3 activation ratios were also fairly straight as indicated by their respective relatively high correlation values (R^2). The correlation values indicate that the pseudo-first order model is not applicable in describing the adsorption of Fe, Ca, Mg and Zn ions onto CPC and CPAC.

Pseudo-second order equation Fitting: The plots in figures. 5-8 show the Fe, Ca, Mg and Zn ions adsorption by CPC and CPAC impregnated at 1:3, 2:3 and 1:1 $ZnCl_2$ activation ratios over the treatment period for second order model adsorption kinetics while Table 5 reveals the values of q_e, K and h_0.

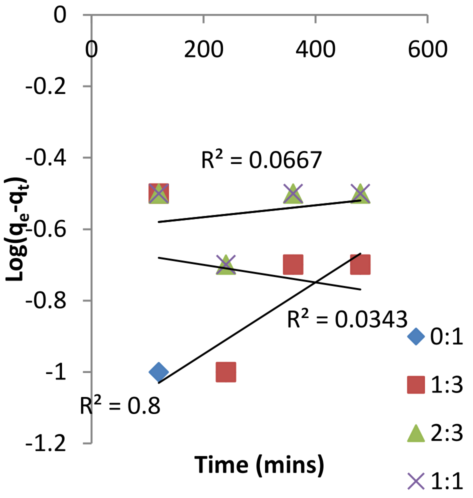

Fig 1: First Order model adsorption kinetics for Fe^{2+}

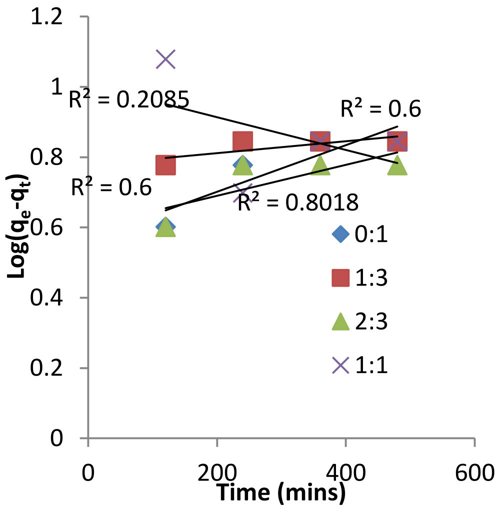

Fig 2: First Order model adsorption kinetics for Ca^{2+}

The plot for both CPC and CPAC for all the metal ions gave a fairly good fit with R^2 values ranging between 0.6 and 0.99, however the trends for Fe adsorption by CPC and CPAC at 1:3 (Figure 5) from the others described. Table 5 also shows that values of k and h_0 for the CPAC at all activation levels were higher than those observed in the CPC also corroborates the fact that activated cassava peel carbon has more pore space after activation thus aiding adsorption. The CPC was also observed to have higher values of q_e for all metal ions adsorbed.

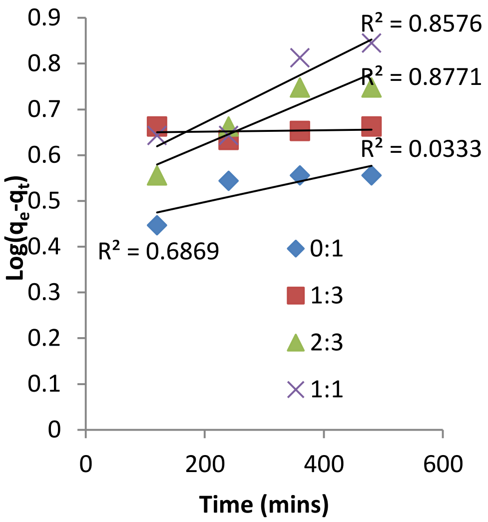

Fig 3: First Order model adsorption kinetics for Mg^{2+}

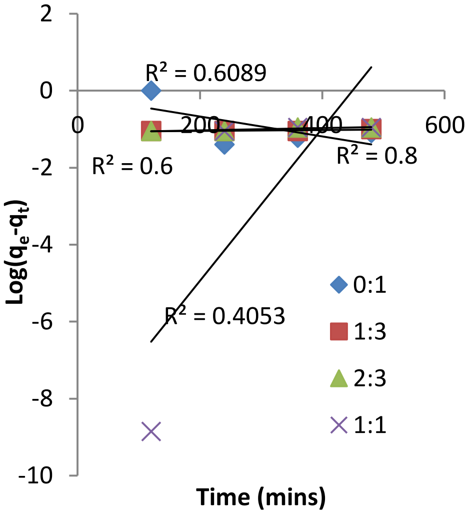

Fig 4: First Order model adsorption kinetics for Zn^{2+}

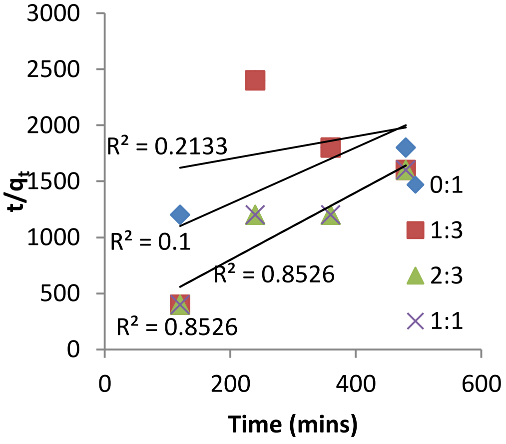

Fig 5: Second Order model adsorption kinetics for Fe^{2+}

This agrees with findings made by Musah *et al.*, (2022) and Alam *et al.*, (2021) which proves that whenever there is an increase in the value of k the adsorption rate will be greater while the observed value for q_e will be lower at when adsorption increases. It then then implies that there is an inverse relationship between q_e values and adsorption rate for most adsorbents.

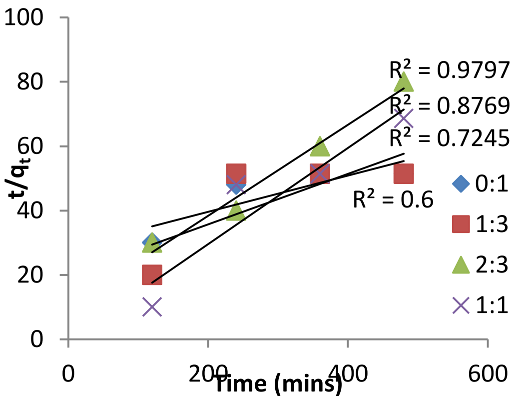

Fig 6: Second Order model adsorption kinetics for Ca^{2+}

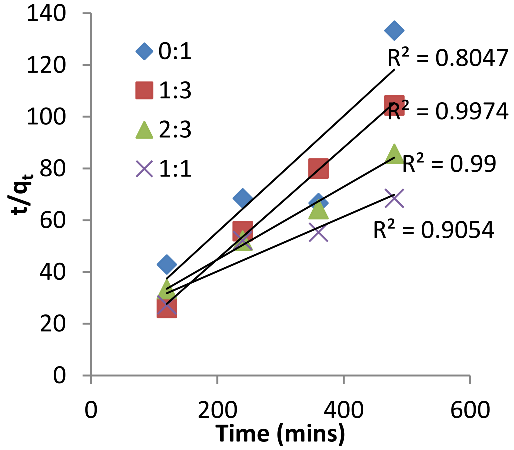

Fig 7: Second Order model adsorption kinetics for Mg^{2+}

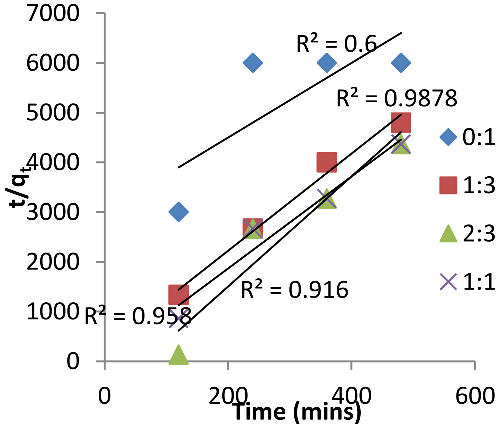

Fig 8: Second Order model adsorption kinetics for Zn^{2+}

Table 5: Pseudo-Second Order Constants

Activation ratio	Constants	Metal ion Fe^{2+}	Ca^{2+}	Mg^{2+}	Zn^{2+}
1:0	K	0	3.66	22.1	0
	h_0	0	2.34 E-1	2.32 E-5	0
	q_e	20000	4043	35.2	0.92
1:3	K	2.33 E2	5.82	28.12	3.92E2
	h_0	123	4.12 E2	2.34	2.12E6
	q_e	500	2030	12.4	0.62
2:3	K	2.71 E1	6.32	29.21	1.33 E-2
	h_0	223	4.08 E2	1.32 E-1	2.44 E2
	q_e	500	2012	10.4	1.53
1:1	K	2.33 E1	6.24	29.18	1.87 E-3
	h_0	142	4.02 E2	22.13	2.21 E1
	q_e	570	103	10.0	2.55

Conclusion: This study has shown that cassava peel carbon can adsorb ions from wastewater. Its also observed that the materials adsorption performance is enhanced when activated with ZnCl2 because of the enlargement of the pore space of the adsorbent. This adsorption characteristics has also been found to be time dependent. The materials adsorption potential was lowest for zinc ion when compared with the other tested ions. The correlation coefficient of Langmuir isotherm of the four metals for CPC and CPAC were between 0.86 to 1, indicating that the adsorbent is very good in adsorption of these metals, however, Freundlich isotherm is found to be unfavorable in describing CPAC's capacity in adsorbing zinc ion contaminant in wastewater. The adsorption of ions by both cassava peel carbon and cassava peel activated carbon materials is best described by the pseudo-second order kinetic model.

Declaration of Conflict of Interest: The authors declare no conflict of interest

Data Availability Statement: Data are available upon request from the first author or corresponding author.

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