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The Removal of Cr^{3+} And Pb^{2+} from Aqueous Solution using Modified Starches from Forest *Anchomanes* (*Anchomanes difformis*)

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ABSTRACT

Water contamination by heavy metals is a global challenge requiring eco-friendly remediation. Progressive discharge of heavy metals in large water bodies has necessitated the need to look for cheap and readily available adsorbents for removal of dissolved metals ions in waters. The potential of modified forest *Anchomanes* starches as adsorbent for the removal of Cr^{3+} and Pb^{2+} are presented in this study. Starch from forest *Anchomanes* tubers was isolated, chemically modified by carboxylation and acetylation processes and characterized with FTIR. On the adsorption of the metal ions, the effects of pH, contact time, starting concentration, and adsorbent dosage were examined. Batch adsorption kinetic and isotherm studies were also done for the uptake. The optimum condition for adsorption were 150 mins, pH 6 and 1.2 g/L adsorbent dosage. The adsorption isotherm studies indicated that the Langmuir model was suitable and appropriate for the adsorption of Cr^{3+} onto acetylated (ACS) adsorbent while Freundlich isotherm model was suitable for the uptake of Cr^{3+} onto carboxymethylated (CMS) adsorbent and Pb^{2+} uptake on both adsorbents. Pseudo-second order kinetics best describe adsorption of Cr^{3+} and Pb^{2+} to ACS whereas, Elovich and Pseudo-first-order kinetic models best describe adsorption of Cr^{3+} and Pb^{2+} on CMS respectively. The results indicate that modified *Anchomanes difformis* starches are effective and efficient in the removal of Cr^{3+} and Pb^{2} in wastewater.

1. INTRODUCTION

Starch is known as a natural, biodegradable material that has been used as adsorbent material in the remediating of contaminants with heavy metals in water (Redha, 2020). It is a polysaccharide molecule made out of the chains of glucose units $(C_6H_{10}O_5)n$, with n ranging from 300 to 1000, bound with a glycosidic linkage (Yu *et al*, 2016). Starch is commonly found in grains (corn, wheat, sorghum, etc.) and tubers like yam, cassava, potato, arrowroot and other root and stem tubers. Due to the heavy dependence on starches from conventional sources for food and non-food applications, other sources are also been sought to reduce the

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overdependence. Starch have been isolated from unconventional sources such as *Chrysophyllum albidum* (Yussuf *et al.,* 2018; Ibikunle *et al.,* 2019) and African bread fruit (Oderinde *et al.,* 2020).

Due to the fact that starch's shape and physiochemical characteristics depend on its organic or biological source, the look, makeup, and characteristics of starch will differ slightly amongst plant sources. (Gebre *et al*, 2006; Neelam *et al,* 2012). Starch in its native forms has limited industrial applications because of its pastes and gels' fragility, fast retrogradation, and insolublity in water at room temperature (Lawal, 2009 Pornsuksomboon, 2016). Chemical modification, improve the functionality of starch and application by introducing additional functional group into the starch structure (Ashogbon and Akintayo, 2014; Ibikunle, *et al,* 2019; Oderinde, *et al,* 2020; Ibikunle, *et al,* 2022; Yussuf *et al.,* 2022). *Anchomanes differmis* commonly known as Forest *Anchomanes* is a wild tuberous plant found in Tropical West Africa, which has so many applications (Akinkurolere & Adedire, 2006: Ahmed, 2018). The starch content of the tuber to is about 28% (Ameen, 2018)

Modern applications of starch as a adsorbent are clearly characterized by its physicochemical properties and dependent on its accessibility requiring little or high calorific value (Omojola *et al,* 2010), therefore, an exhaustive assessment of the vital parameters is significant in its industrial use. The aim of this study is to apply modified starches obtained from forest *Anchomanes* for the adsorption of Cr^{3+} and Pb^{2+}.

2. MATERIALS AND METHOD

2.1.Collection of Starch Samples

Forest *Anchomanes* (*Anchomanes differmis*) tuber was collected at the permanent site of Olabisi Onabanjo University Ago-Iwoye, Ogun state, and authenticated at the Department Plant Science of the University. The sample was then taken to the laboratory for further analysis.

2.2.Isolation of Forest Anchomanes Starch

The tubers of Forest *Anchomanes* were chopped into 2 cm long pieces after being peeled and cleaned. 500 g of the chips and some water were blended into a homogenous mixture. After being combined with several times its original amount of water, the resulting slurry was sieved through muslin cloth. After letting the starch milk settle for the entire night, the supernatant was decanted. Distilled water was used to wash this starch multiple times. After being air-dried, ground, weighed, and kept in a plastic container, the resulting off-white starch (Nwokocha *et al*, 2011).

2.3.Preparation of Carboxymethylated Starch (CMS)

20 g of NaOH was dissolved in 30 mL of water in a 400 mL beaker. 200 mL of Isopropanol was added to the solution to give solvent to water ratio of 1: 0.15. 40 g Forest *Anchomanes* starch was added to the mixture with continuous stirring for 1 hour. 30 g of monochloroacetic acid was added and the mixture was further stirred for 1.5 hrs. The mixture was then allowed to settle and the supernatant was decanted. The slurry was washed with methanol, dispersed in acetone for 20 mins, filtered and dried in an oven at 40°C for 4 hr (Lawal *et al*., 2007).

2.4.Preparation of Acetylated Starch (ATS)

70 g starch was dispersed in 350 mL distilled water, stirred magnetically for 20 mins and maintained at a constant temperature. NaOH (1 M) was added in dropwise to adjust the pH to 8.0, the mixture was then gradually mixed with 10 mL of acetic anhydride over the course of an hour, keeping the pH between 8.0 and 8.5. The addition of acetic anhydride was followed by a 5-minute reaction period. Using 0.5 M HCl, the slurry's pH was eventually brought to 4.5. After that, it was filtered, cleaned three times with distilled water, and allowed to air dry for 48 hrs at room temperature (Lawal, 2004).

2.5.Characterization of the Forest Anchomanes Starch

Characterization was done using Fourier Transform Infrared Spectrometer (FTIR) to identify the functional group.

2.6.Batch Adsorption Study

The adsorption investigation was carried out in a glass tube at room temperature using batch trials with various parameters (Gabriel *et al*, 2015). The metal ions in the filtrate were then analyzed with Atomic Absorption Spectrophotometer (AAS) (Haware and Pramond, 2011). Equation 1 was utilized to determine the quantity of metal ions adsorbed from the solution.

$$P = \frac{C_i - C_f}{C_i} \times 100 \qquad 1$$

where P = Percentage ion removed; C_i and C_f = Initial and final concentrations (mg/L).

2.7. Determination of Optimum Condition for Adsorption

The effect of pH, contact time, adsorbent dosage and initial metal ion concentration were determined using the method of Babalola *et al.* (2011). For the effect of pH on metal adsorption, 20 mL of standard solutions were poured into 5 conical flasks for each metal solution. The pH of the samples was adjusted to between 2 to 10 using 0.1 M NaOH and 0.1 M HCl. Subsequently, 0.2 g of the modified starches were added to each flask and agitated for 1 hr using orbital shaker at 150 rpm. The samples were then filtered and the metal concentrations present were analyzed using AAS. The optimum pH for Cr^{3+} and Pb^{2+} adsorption was ascertained from maximum percentage adsorption. The pH obtained was then used for other parameters.

2.8.Adsorption Isotherms

Nimbofa *et al*. (2017) method was adopted for adsorption isotherms. The stock solution was used to prepare metal ion solutions (20–100 ppm). After 20 mL of each of these solutions' samples were transferred into various conical flasks, the initial pH was adjusted to the ideal level for each metal. Then, each flask received 0.2 g of CMS and ACS individually. After 2 hrs of agitation with an orbital shaker set to 150 rpm, the contents of the flask were centrifuged for fifteen minutes at 8000 rpm. Each flask's contents was filtered, and an AAS analysis was used to ascertain how much heavy metal was present in the filtrate. Equation 2.0 was used to determine the modified starches' adsorption capacity.

$$Q = \frac{V(C_I - C_e)}{m} \quad (2)$$

Where V is the volume of solution (mL), m is the mass of adsorbent (g), Q is the adsorption capacity of the modified starch (mg/g), Ci and Ce are the initial metal concentration (mg/L), and metal concentration at equilibrium (mg/L) respectively.

3. RESULTS AND DISCUSSION

3.1.Percentage Yield and Characterization

The percentage yield of starch from *Anchomanes difformis* was 64.65% of its biomass. This is higher than the 28% reported by Ameen (2018). This may be attributed to the painstaking procedure involved in the isolation process. The results of Fourier Transforms Infrared Spectrophotometer (FTIR) analysis show vibrational frequencies of functional groups present in ACS and CMS before and after metal adsorption. Figure 1 shows the FTIR spectra before adsorption with broad band observed around 3338 cm^{-1} indicating the presence of an O-H stretch (Pons *et al*, 2004). The peaks observed at 2907cm^{-1} and 2163 cm^{-1} could be attributed to sp^3 C-H and C-C respectively, while the band at 1053 cm^{-1} could be attributed to alcohol C-O stretch (Sheng *et al*, 2004). Furthermore, Figure 2's shift in absorbance peak frequency suggests that metal binding processes were occurring on the adsorbent's active sites (Sheng *et al*, 2004).

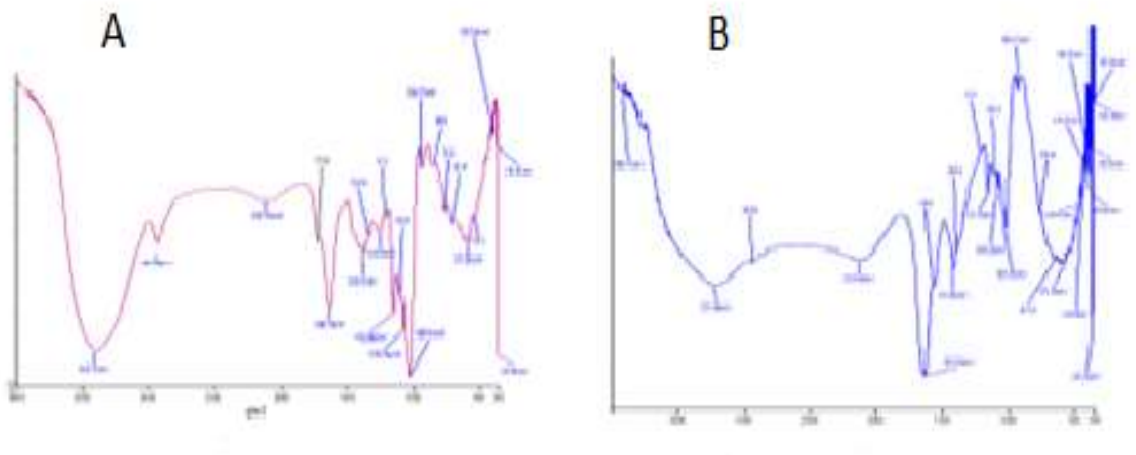

Figure 1: FTIR Spectra of (A) ACS and (b) CMS before metal adsorption

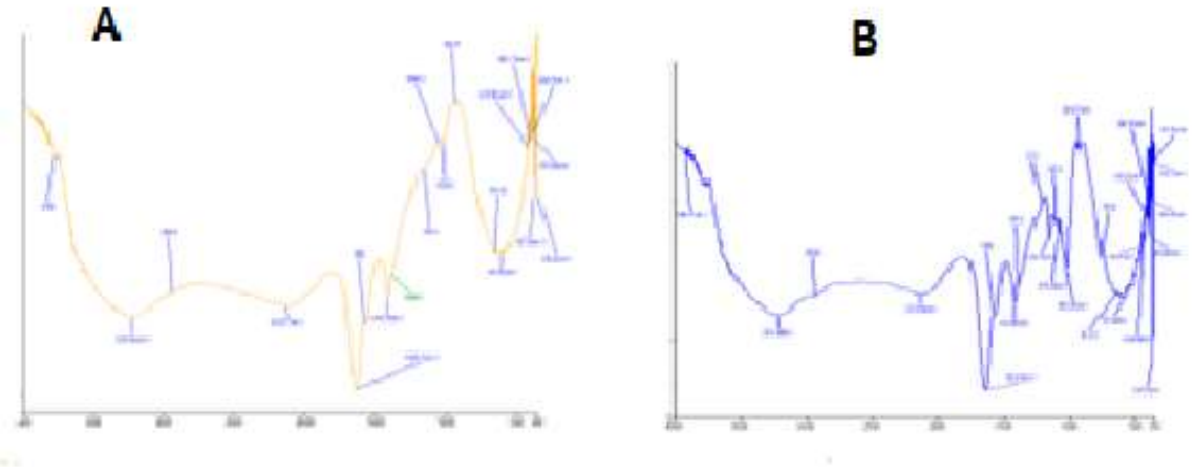

Figure 2: FTIR Spectra of (A) ACT and (b) CB after metal adsorption

3.1.1. Adsorptions Studies

Effect of pH

Figure 3 displays the findings of CMS and ACS's investigation into how pH affects the adsorption of Cr^{3+} and Pb^{2+}. When pH rises, more deposited metal ions are absorbed. For both metals, the highest adsorption happens at pH 6, when Cr^{3+} is removed at a rate of 80.28% and Pb^{2+} at a rate of 74.46%. The amount of adsorbed metal ion either stays constant or significantly drops above pH 6. This demonstrates that the starch's surface charge is pH dependant due to its varied surface charge features. Divalent metal retention on surfaces by adsorption, inner sphere surface complexation, and precipitation has been found to increase with increasing pH (Apell and Lena, 2002; Lingamdinne ,2017).

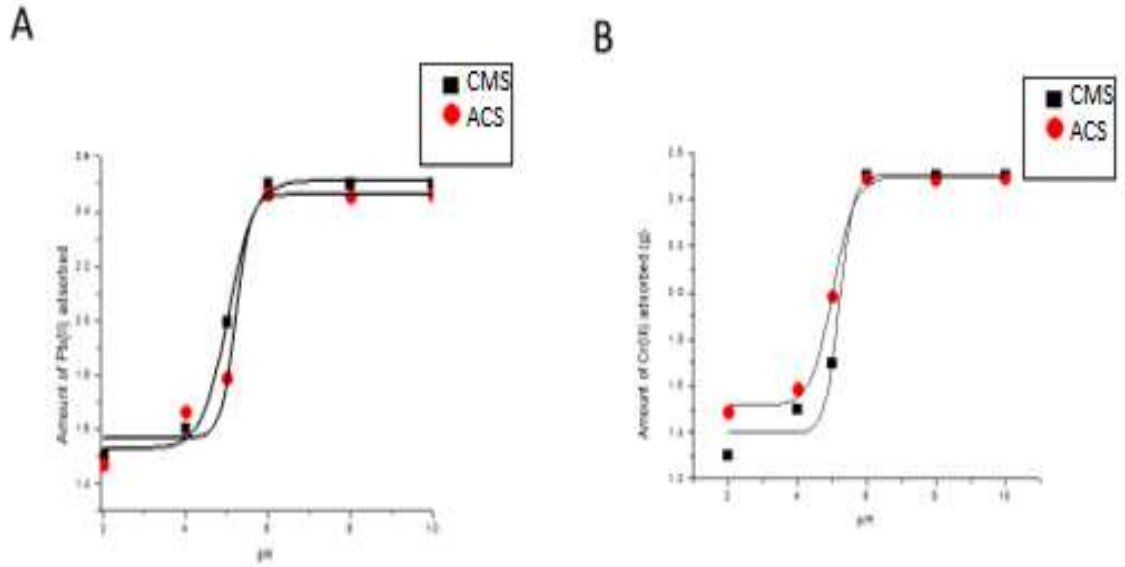

Figure 3: Effect of pH on adsorption of (A) Cr^{3+} and (B) Pb^{2+}

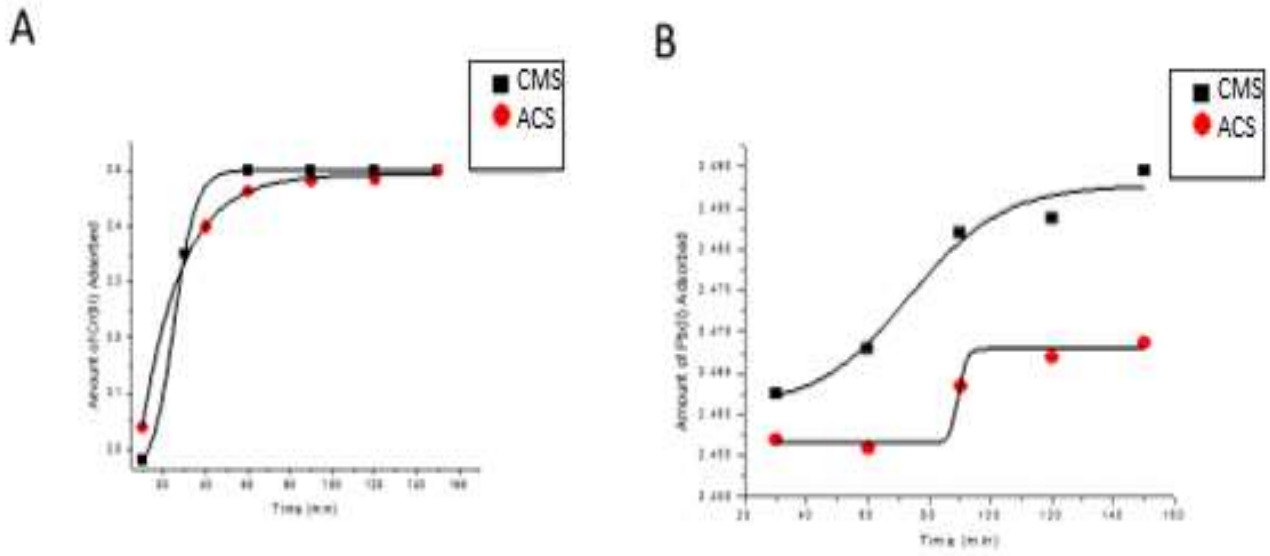

Figure 4: Effect of contact time on adsorption of (A) Cr^{3+} and (B) Pb^{2+}

Effect of Contact Time

Figure 4 displays the findings of CMS and ACS's investigation into the impact of contact time on the adsorption of Cr^{3+} and Pb^{2+}. It is clear from the data that as contact time increases, so does the elimination of metal ions. Nonetheless, for both metal ions, the percentage of elimination achieved equilibrium in less than 150 minutes. Babalola *et al*., (2011) reported a similar finding about the adsorption of Cd^{2+} by Sodom apple leaves. The availability of a high number of active sites may have contributed to the first significant increase in the rate of adsorption, which decreased progressively towards equilibrium (Mustapha *et al.*, 2023)

Effect of Adsorbent Dose

Figure 5 illustrates how the adsorbent dosage (0.2 g - 1.4 g/L) affects the metal ion adsorption. Both metals show a general increase in percentage removal, with maximum adsorption seen at 1.2 g. Subsequent increases in the dose of adsorbent did not result in an increase in adsorption. When the amount of adsorbent is raised beyond 1.2 g/L, there will be less metal ions available for adsorption per gram of adsorbent. Their different chemical affinities and ion exchange capacities with regard to the chemical functional group on the adsorbent surface may also be the cause of the variation in adsorption capacity (mg/g) at the same initial metal ion concentration and contact time. Thus, greater adsorbent surface area and the availability of additional adsorption sites are responsible for the increase in adsorption with adsorbent dose (Tamilselvan, 2011).

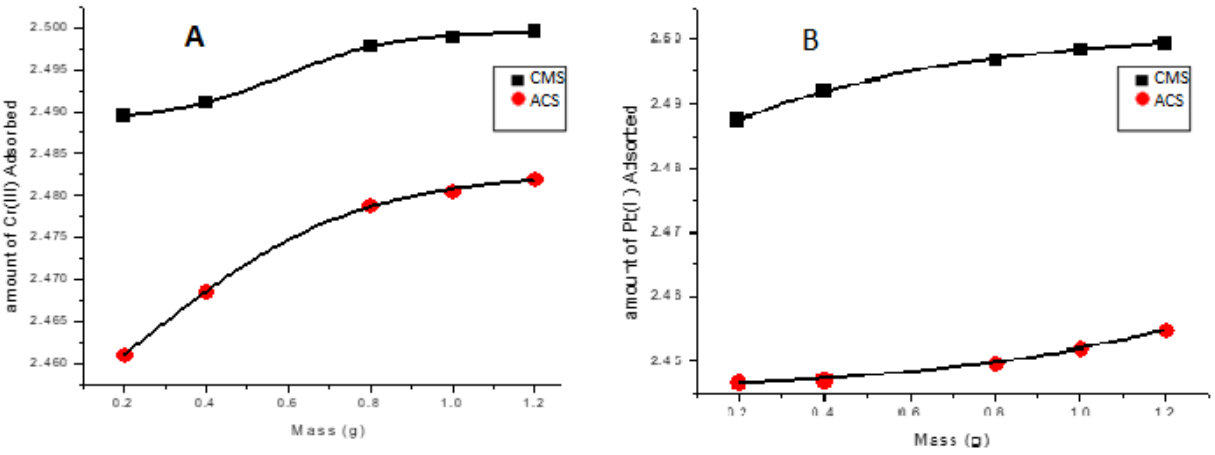

Figure 5: Effect of initial adsorbent dosage on adsorption of (A) Cr^{3+} and (B) Pb^{2+}

Effect of initial Metal ion Concentration

Figure 6 illustrates how changing the starting metal concentration (10–100 mg/L) affects the removal of metal ions. The findings demonstrate that as initial metal ion concentration rises, so does adsorption capacity. As the initial concentration rises, the electrostatic contact between the metal ions and the adsorbent active sites gradually develops, resulting in a steady increase in adsorption capacity (Shi *et al.*, 2009). Furthermore, a rise in metal concentration enhances total mass transfer to the adsorbent's surface (Babalola *et al.*, 2011; Mustapha *et al.*, 2023).

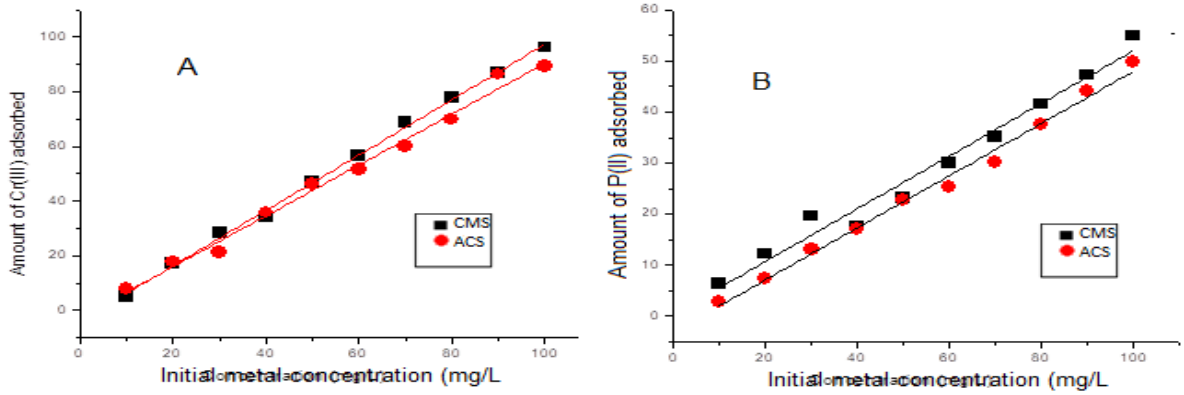

Figure 6: Effect of metal concentration on adsorption of (A) Cr^{3+} and (B) Pb^{2+}

3.1.2. Adsorption Isotherms

The equilibrium between the concentration of the dissolved adsorbate and the amount of adsorbate that builds up on the adsorbent was described using the adsorption isotherms. Adsorption models based on Freundlich and Langmuir isotherms were used to the obtained experimental data. The Langmuir isotherm, which shows the equilibrium distribution of metal ions between the solid and liquid phases, is based on the theoretical

idea that there is only one adsorption layer on an adsorbent. Equation 3.0 represents the Langmuir equation.

$$1/Q_e = 1/Q_m + 1/(K_L C_e Q_m) \quad (3)$$

Where Q_e is the amount of adsorbate adsorbed at equilibrium (mg/g), K_L is the Langmuir constant related to the energy of adsorption (L/mg), Q_m is the maximum sorption capacity corresponding to complete monolayer coverage (mg/g), and C_e is the equilibrium solute concentration (mg/L).Q_m and K_L are obtained from the intercept and slope of the plot of $1/Q_e$ and $1/C_e$. The results of obtained by fitting the experimental data of metal ions adsorption to the Langmuir equation are shown in figure 7.

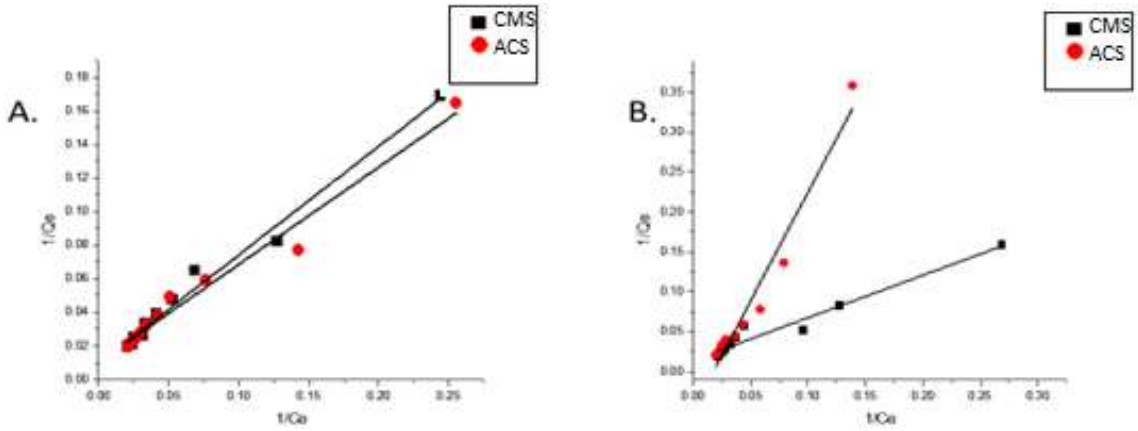

Figure 7: Langmuir Isotherm for the adsorption of (A) Cr^{3+} and (B) Pb^{2+}

Equation 4.0 provides the logarithmic form of the Freundlich isotherm, which assumes heterogeneous surface energies.

$$\log Q_e = \log K_f + 1/n\,(\log C_e) \quad (4)$$

where Kf and n are Freundlich constants that, respectively, represent the maximum adsorption intensity and capacity. Figure 8 displays the outcomes of applying the Freundlich equation to the experimental data.

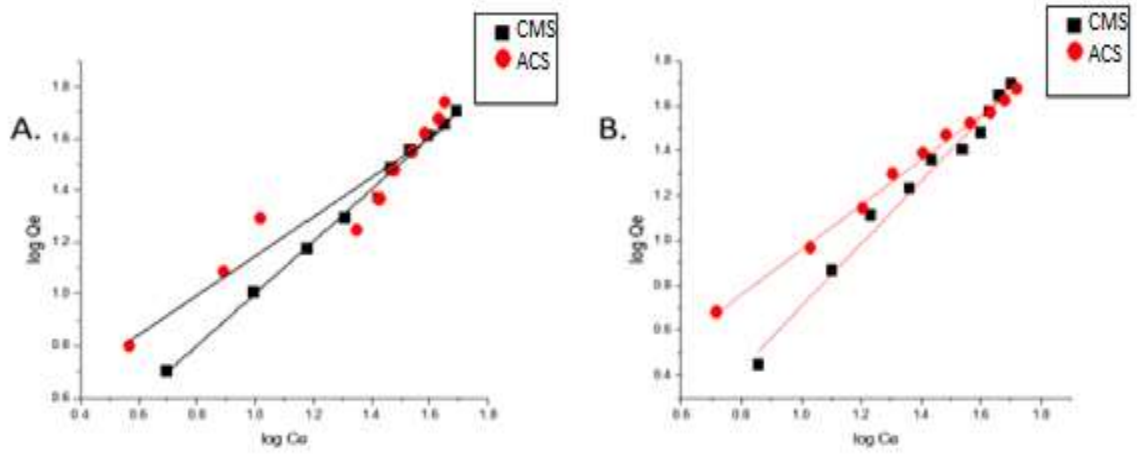

Figure 8: Freundlich Isotherm for the adsorption of (A) Cr^{3+} and (B) Pb^{2+}

Table 1: Isotherm Parameters for Adsorption of Cr^{3+} and Pb^{2+} using Modified Starch at 100 mgL^{-1} as obtained from the plot graph in Figure 7 and Figure 8 respectively.

Isothermal model	**Modification**	**Parameter**	**Cr^{3+}**	**Pb^{2+}**
Langmuir	CMS	Q_e(mgg^{-1})	4.72	0.49
		K_L (min^{-1})	0.56	0.43
		R^2	0.9720	0.9479
	ACS	Q_e(mgg^{-1})	4.77	3.83
		K_L (min^{-1})	0.55	3.92
		R^2	0. 9825	0.9547
Freundlich	CMS	Q_e(mgg^{-1})	3.32	2.34
		Kf (min^{-1})	3.63	2.74
		R^2	0. 9948	0.9842
	ACS	Q_e(mgg^{-1})	3.38	2.55
		Kf (min^{-1})	3.44	2.51
		R^2	0.8946	0.9951

The adsorption isotherm studies indicated that the Langmuir model was progressively suitable and appropriate for the adsorption of Cr^{3+} on ACS than the Freundlich model as a result of the higher correlation coefficient as shown in Table 1. This suggest that adsorption of Cr^{3+} occurs via monolayer chemisorption at specific homogeneous sites on the adsorbent (Abdunnaser *et al*,2014; Lingamdinne *et al.*, 2017). However, the Freundlich isotherm gives the best fit for the adsorption of Pb^{2+} for both ACS and CMS as well as Cr^{3+} for CMS. Freundlich isotherm model has been used to describe Pb^{2+} uptake from yeasts biomass above 10°C (Ferraz and Teixeira, 1999) as well as Cr^{3+} (Brum *et al*, 2010; Tamilselvans *et al*, 2011).

3.1.3. ***Adsorption Kinetics Study***

Based on the linear forms of the model's equation as given in equations 5-7, kinetic data were fitted using pseudo-first order, pseudo-second order, and Elovich kinetic models to explore the mechanism of adsorption and potential rate controlling stages. Whether or not a model adequately describes the adsorption process is determined by how linear each model is when plotted, as seen in figures 9–11.

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303}t \qquad 5$$

where qe is the mass of metal adsorbed at equilibrium (mg/g), q_t the mass of metal adsorbed at time t (min) and k_1 is the pseudo-first order rate constant.

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t \qquad 6.0$$

Where k_2 is the pseudo-second order rate constant of adsorption (mg/g.min)

$$q_t = A + B\ln t \qquad 7$$

Where A and B are the Elovich constants corresponding to the extent of surface coverage and rate of sorption at zero coverage respectively.

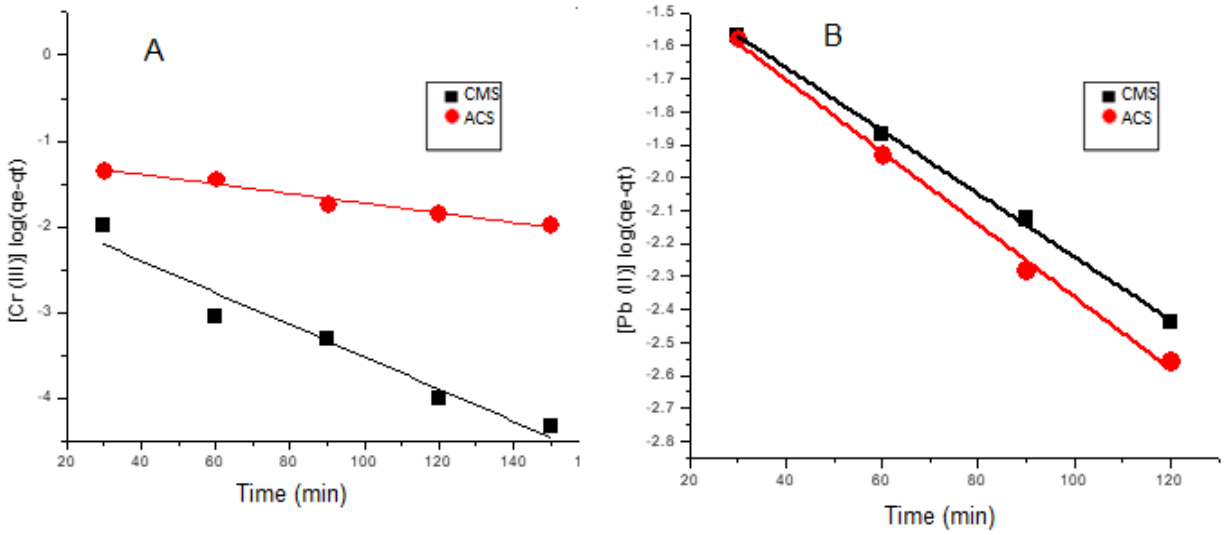

Figure 9: Pseudo-first-order plot for the adsorption of (A) Cr^{3+} and (B) Pb^{2+}

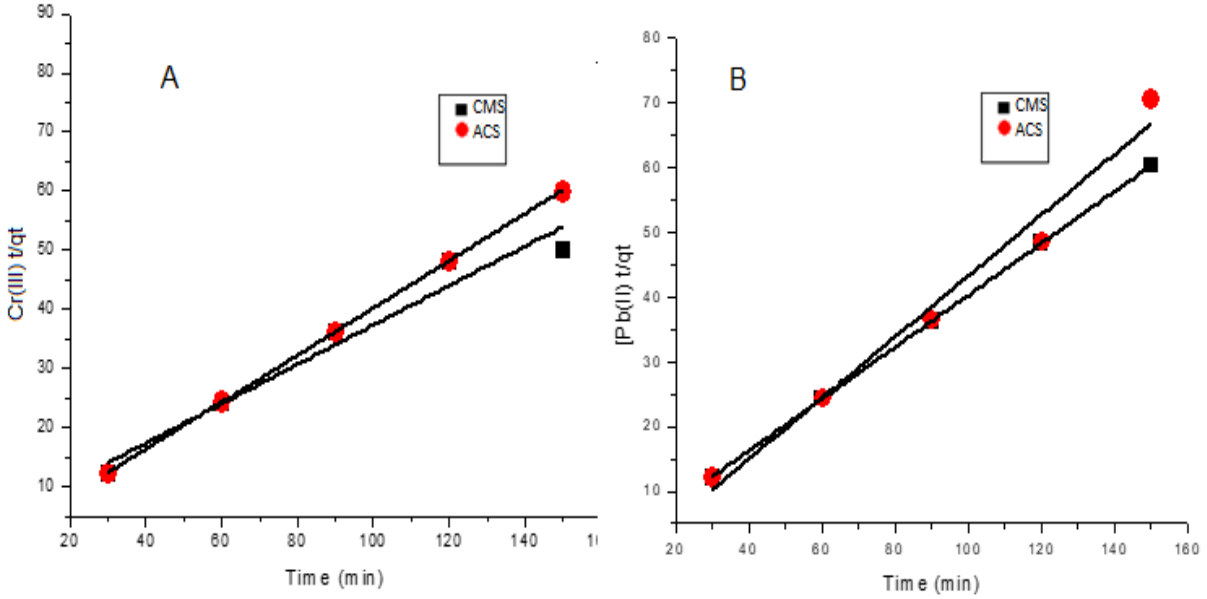

Figure 10: Pseudo-second-order plot for the adsorption of (A) Cr^{3+} and (B) Pb^{2+}

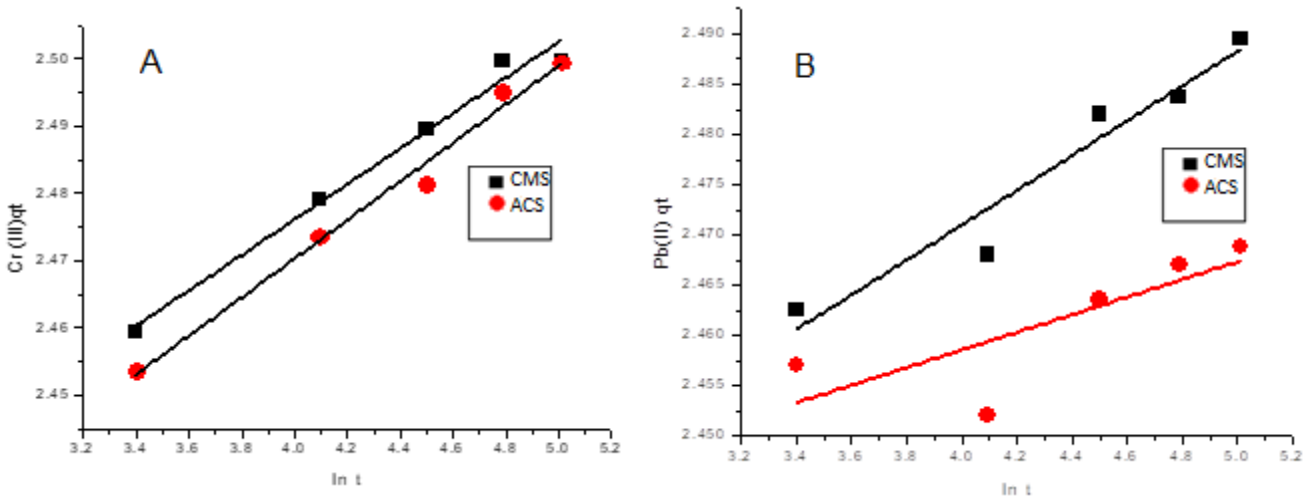

Figure 11: Elovich plot for the adsorption (A) Cr^{3+} and (B) Pb^{2+}

This study reveals that the most suitable kinetic model in describing the adsorption of Cr^{3+} onto CMS is the Elovich while the Pseudo-second Order best describe its adsorption onto the ACS. Similarly, for the adsorption of Pb^{2+} to the CMS adsorbent, Pseudo-first order model gives the best fit with the experimental data, while the Pseudo-second order model is the most suited for its adsorption onto the surface of ACS based on the correlation coefficients obtained as presented in table 2. According to the pseudo-first-order model, the number of vacant sites determines how quickly an adsorption site is occupied (Ertugay and Bayhan. 2008; Babarinde *et al*, 2013). Contrarily, the Pseudo-second order model is based on the assumption that adsorption is a result of chemisorption process occurring between the adsorbates and functional groups on the surface of adsorbents (Mustapha *et al*, 2023). Additionally, the Elovich adsorption is believed to proceed via a number of mechanisms (Ogundiran, 2021).

4. CONCLUSION

The primary focus of this study was the removal of Cr^{3+} and Pb^{2+} from aqueous solution using an inexpensive and efficient adsorbent. An appreciable yield (≈65%) of starch was obtained from forest *Anchomanes* which was successfully modified to its derivatives. The adsorption of Cr^{3+} and Pb^{2+} by CMS and ACS was maximum at pH 6, 1.2 g/L dosage and was completed in 150 mins. The Kinetic studies showed adsorption of Cr^{3+} and Pb^{2+} to ACS followed pseudo-second order kinetics, while Elovich and Pseudo-first-order controlled the adsorption of Cr^{3+} and Pb^{2+} onto CMS respectively.

Table 2: Kinetic parameters for the adsorption of Cr^{3+} and Pb^{2+} as obtained from the plots in in figure 9-11

Kinetic model	**Parameter**	**Cr^{3+}**	**Pb^{2+}**
Pseudo-first order	$Q_e(mgg^{-1})$	81.44.	80.26
CMS	k_1 (min^{-1})	1.2 x 10^{-2}	1.6 x 10^{-2}
	R^2	0.9356	0.9952
ACS	$Q_e(mgg^{-1})$	82.14	84.02
	k_1 (min^{-1})	1.2 x 10^{-2}	1.6 x 10^{-2}
	R^2	0.9986	0.9979
Pseudo-second Order	q_e, cal(mgg^{-1})	42.51	53.87
CMS	k_2(g $mg^{-1}min^{-1}$)	1.07 x 10^{-2}	1.18 x 10^{-2}
	R^2	0.9488	0.9740
ACS	q_e, cal(mgg^{-1})	42.51	53.87
	k_2(g $mg^{-1}min^{-1}$)	1.07 x 10^{-2}	1.18 x 10^{-2}
	R^2	0.9999	0.9998
Elovich	A	38.97	41.46
CMS	B	25.13	23.73
	R^2	0.9838	0.9170
	A	38.97	41.46
ACS	B	25.13	23.73
	R^2	0. 9792	0. 5090

The Langmuir adsorption isotherm suggest a monolayer chemisorption mechanism for the adsorption of Cr^{3+} onto specific homogeneous sites on acetylated forest anchomonas starch. On the contrary, the adsorption of Pb^{2+} onto both modified starch adsorbent and Cr^{3+} onto carboxymethylated starch is best described by the Freundlich isotherm model. Therefore, modified forest *Anchomanes* tuber starch has a lot of potential for use as an adsorbent to remove effluent containing Pb^{2+} and Cr^{3+}.

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