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Removal of chromium and nickel ions from aqueous solution by adsorption on modified coconut husk

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The study investigated the adsorption of Cr(VI) and Ni(II) on modified coconut husk (low cost adsorbent) for the remediation of Cr(VI) and Ni(II) from some synthetic industrial waste effluents. The coconut husk was modified by mixing with 0.1 M NaOH and HCl respectively. The effects of varying adsorbent loading, hydrogen ion concentration, contact time and temperature of adsorption were studied. Similar experiments were carried out using unmodified coconut husk, in order to compare the results obtained from the modification experiments. The result showed that the HCl modified adsorbent gave $96\% \pm 3.6$ removal for Cr(VI) at a pH of 4.0 while about $99\% \pm 0.7$ of Ni(II) was removed at a higher pH of 7.0. The unmodified adsorbent gave a $90\% \pm 8.8$ adsorption for Cr(VI) and $97\% \pm 1.5$ for Ni(II) respectively. However, the NaOH modified adsorbent gave the least adsorption of $40\% \pm 9.9$ for Cr(VI) and $80\% \pm 6.1$ for Ni(II). The equilibrium data for the adsorption of Cr(VI) and Ni(II) on coconut husk was tested using both the Freundlich and Langmuir adsorption isotherms. The Freundlich isotherm was found to be more suitable for Cr(VI) adsorption, while the Langmuir isotherm was observed to better for Ni adsorption on coconut husk. To evaluate the mechanism of adsorption, pseudo-first order and second order kinetic models were used. The adsorption mechanism was found fit the second order. The HCl modified adsorbent gave the best result for removal of Cr(VI) from synthetic industrial effluents.

Keywords: Heavy metals, low cost adsorbents, remediation, waste water.

INTRODUCTION

Heavy metals are usually present in wastewaters which are released into the environment from various industries. The adverse effects caused by these heavy metals are of great environmental concern. Heavy metals are non biodegradable and accumulate in living organisms thereby causing various diseases and disorders (Bailey et al., 1999). Chromium and nickel are frequently used in industrial processes such as metal plating industries, galvanizing industries, mining operations and tanneries and are usually present in high concentrations in the liquid wastes which are released directly into the environment without any pre-treatment. Once in the environment, chromium exists mainly in two oxidation states (Cr(III) and Cr(VI)). While Cr(III) is relatively innocuous, Cr(VI) is toxic, carcinogenic and mutagenic. It is highly mobile in soil and aquatic system and is a strong oxidant capable of being adsorbed by the skin (Singh and Singh,

2002). Nickel toxicity has been known to inhibit spermatogenesis, amylase enzymes, insulin formation and kidney formation. Due to the high toxicity of these metals, there is increasing interest in the development of techniques for their removal from wastewaters before they are disposed into the environment.

Some of the techniques which have been used in the removal of metals from effluents include ion-exchange, chemical precipitation, electro dialysis, electrolytic extraction, reverse osmosis and cementation. These methods are expensive and have the inability to remove metals at low concentration (Faur-Brasquet et al., 2002; Bishnoi et al., 2004). Compared to the aforementioned techniques, adsorption has shown to be a more economically favourable alternative for the removal of metals from aqueous solutions (Namasivayam et al., 1999). The most widely used adsorbent is the activated carbon. Activated carbon cloths have been used as adsorbent in the removal of metals in both monocomponent and multicomponent solutions (Zhanpeng et al., 1991; Gabaldon et al., 1996; Madukasi et al., 2001) and it has been shown that it has an adsorption kinetics which is about two to 20 times grea-

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ter than those of granular activated carbon (Faur-Brasquet et al., 2002).

Due to the relatively high cost of activated carbon, cost effective adsorbents for treatment of metal contaminated wastewaters are necessary particularly in developing economies like Nigeria. Some agricultural waste products have been shown to have the potential as inexpensive sorbents. Due to their low cost, after these materials have been expended, they can be disposed of without expensive regeneration (Bailey et al., 1999). Some studies on the use of adsorbent such as sawdust and teak tree bark, waste tea, coconut shell and dry plants on the removal of metals from aqueous solutions have been reported (Olayinka et al., 2005; Olayinka et al., 2007; Benhima et al., 2008). The adsorption capacities of these materials have been shown to be dependent on experimental conditions such as pH, metal concentration and adsorbent loading.

The aim of this research is to investigate the use of modified coconut husk in the removal of metals from aqueous solutions. Coconut husk is a low cost adsorbent which is a waste material from coconut. Coconut is abundant in Nigeria and has a high sorption capacity due to its high tannin content. The research involved the improvement of the sorption capacity of this low cost adsorbent by modification with HCl and NaOH respectively. The study also involved the examination of experimental conditions such as pH of the solution, temperature, contact time and adsorbent loading on the removal of Cr(VI) and Ni(II) from aqueous solutions. The Freundlich and Langmuir adsorption isotherms were used to investigate the adsorption process. Kinetic study was also carried out to evaluate the order of adsorption.

MATERIALS AND METHODS

Adsorbents

The adsorbent was prepared as described by Hanafiah et al. (2006). The coconut husk was cut into small pieces and blended, washed with distilled water to remove dirt and colour and air-dried for 24 h to avoid thermic deactivation of the adsorbent surface. It was sieved to pass through a 2 mm stainless steel endecott sieve and a portion of this was stored in clean polyethylene containers (labeled as unmodified adsorbent) prior to analysis. For the modification of the adsorbent, about 400 g of the washed adsorbent was mixed with 600 ml of 0.1 mol dm⁻³ NaOH. The mixture was heated at 120°C for 30 min with occasional stirring. The powdered coconut husk was separated using a Buckner funnel and a vacuum pump and washed with distilled water until the washings were free of color and the pH of wash solution was 7. The washed adsorbent was air-dried for 24 h and labeled as NaOH- modified coconut husk. The HCl modification was also carried out using the same procedure as the NaOH modification but with 0.1 mol dm⁻³ HCl.

The concentrations of Cr and Ni in the adsorbent were determined by placing 1g of the adsorbent in 10 ml deionized water for one hour with continuous agitation, after which it was centrifuged at 2000 rpm with a Remi R-8C laboratory centrifuge. The supernatant was carefully decanted into acid cleaned polyethylene containers and analysed using a flame Atomic Absorption Spectrophotometer (FAAS) Perkin Elmer Analyst 200. The pH of the absor-

Table 1. Main characteristics of adsorbates.

Property	Cr(VI)	Ni(II)
Atomic weight (g mol ⁻¹)	51.996	58.693
Formular	K ₂ Cr ₂ O ₇	NiCl ₂ .6H ₂ O
Solubility @ 20°C (g cm ⁻³)	11.5	2.54
Ionic radius (Å)	0.44	0.69
Pauling Electronegativity	1.66	1.91

bents used (modified and unmodified) was determined according to British Standard ISO 10390 (2005), using a Mettler Toledo pH meter.

Adsorbates

The solutions of Cr(VI) and Ni(II) metal ions were prepared from analytical grade K₂Cr₂O₇ and NiCl₂ respectively. 1,000 mg/L aqueous solutions (stock solutions) of these salts were prepared with deionized water in 1% HNO₃ solution and these stock solutions were diluted with deionized water to obtain the working standard solutions. The main characteristics of these ions are summarized in Table 1.

Adsorption experiments

For the adsorption experiment, the effect of adsorbent loading on the adsorption of the ions was investigated. 0.2- 1.0 g of the adsorbent was weighed respectively into conical flasks. 20 ml of 10 mg/L solution of each of the metal ion solution was added and the mixture shaken with an IKA HS 260 basic reciprocating shaker at 150 rpm for 30 min. The mixtures were centrifuged at 2,000 rpm for 30 min, the supernatant decanted and the metal ion content was determined using a Perkin Elmer AA Analyst 200 Flame AAS. The % adsorption was determined by

$$\frac{C_o - C_a}{C_o} \times 100$$

Where; C_o = Initial concentration of solution, C_a = Concentration of the solution after adsorption

In order to investigate the effect of concentration on the adsorption of metal ions, 0.4 g of the adsorbent was added to 20 ml each of varying concentrations (between 10 - 100 mg/L) of the metal ion solutions. The mixtures were shaken, centrifuged and the concentration of the metal ions adsorbed was determined. The effect of contact time was also investigated by adding 0.4 g of the adsorbent to 20 ml of 10 mg/L and shaking using varying contact times (between 30 – 180 min) and the percentage of adsorbed ions determined. The effect of pH on adsorption of the metals was investigated using 0.4 g of the adsorbent and 20 ml of 10 mg/L and the pH of the solution was adjusted between 2 - 12. The mixture was shaken for 90 min, centrifuged and the amount of ion adsorbed was determined.

In order to investigate the effect of modifying the adsorbent, each of the experiments were repeated using NaOH-modified coconut husk and HCl-modified coconut husk. For quality control, the experiments were carried out in triplicates.

Adsorption Isotherm models

The Freundlich and Langmuir adsorption isotherm models were

used. The Freundlich isotherm (Freundlich, 1906) is expressed as

$$N_e = K_f C_e^{1/n}$$

Where; $N_e = x/m$ (amount of metal ions per unit mass of adsorbent) when the equilibrium concentration is C_e

To simplify the equation, it was linearized thus:

$$\log N_e = \log K_f + 1/n \log C_e$$

The Freundlich Isotherm Coefficients were determined by plotting $\log N_e$ against $\log C_e$. The Freundlich equation is an empirical equation based on adsorption on a heterogeneous surface. The constants indicate the adsorption capacity and the adsorption intensity.

The Langmuir isotherm (Langmuir, 1918) is based on the assumption that adsorption does not proceed beyond monolayer coverage and that all sites available on the adsorbent surface are equivalent and the surface is perfectly uniform. It also assumes that there is no interaction between adjacent adsorbed molecules.

$$\theta = \frac{N_e}{Q_m} = \frac{bC_e}{1 + bC_e}$$

Where; b , is the adsorption equilibrium constant ($l \text{ mg}^{-1}$) that is related to the apparent energy of adsorption and Q_m is the quantity of adsorbate required to form a single monolayer on unit mass of adsorbent (mg g^{-1}) and N_e is the amount adsorbed on unit mass of the adsorbent (mg g^{-1}) when the equilibrium concentration is C_e (mg/L). The equation can be linearized thus

$$\frac{C_e}{N_e} = \frac{1}{bQ_m} + \left(\frac{1}{Q_m} \right) C_e$$

A plot of (C_e/N_e) vs C_e should yield a straight line if the Langmuir equation is obeyed by the adsorption equilibrium (Gupta and Babu, 2009)

Adsorption kinetics

The mechanism of the adsorption of Cr(VI) and Ni(II) can be explained using pseudo first-order and second-order kinetic models.

Pseudo first-order kinetics

The non-linear form of pseudo first-order equation is given by

$$\frac{dN_t}{dt} = k_{ad} (N_e - N_t) \quad (\text{Ho and Mckay, 1999})$$

Where N_e and N_t are the amounts of each of Cr(VI) and Ni(II) adsorbed at equilibrium time and any instant of time (t) respectively. k_{ad} is the rate constant. The integrated rate law then becomes

$$\log(N_e - N_t) = \log N_e - \frac{k_{ad} t}{2.303}$$

Plot of $\log(N_e - N_t)$ vs t gives a straight line and the adsorption rate constant k_{ad} can be computed

Second-order kinetics

Applicability of the second order kinetics is tested with the rate equation

$$\frac{dN_t}{dt} = k_2 (N_e - N_t)^2$$

Where; k_2 is the second order rate constant. From the boundary conditions $t = 0$ to $t = t$ and $N_t = 0$ to $N_t = N_t$, the integrated rate law becomes

$$\frac{1}{(N_e - N_t)} = \frac{1}{N_e} + k_2 t$$

this can be rewritten as:

$$\frac{t}{N_t} = \frac{1}{h} + \left(\frac{1}{N_e} \right) t$$

Where; h = is the initial sorption rate as $t \rightarrow 0$. The plot of $t/N_t =$ gives a linear relationship and N_e , k_2 and h . (Ho and Mckay, 1999; Gupta and Babu, 2009)

RESULT AND DISCUSSION

Effect of adsorbent loading on the adsorption of Cr and Ni

The result for the adsorption of Cr and Ni is shown in Figure 1. For Cr, HCl-modified adsorbent removed almost 100% of the metal from the synthetic effluent regardless of the adsorbent loading. This could be due to the fact that HCl-modified adsorbent has positively charged sites due to the acid and the Cr(VI) which is being adsorbed is an anion. The presence of a higher number of adsorption sites on even a small amount of the adsorbent probably accounts for the reason why variation in adsorbent loading had no effect since 0.2 g of adsorbent already removed almost 100%.

The % adsorption of Cr by the unmodified adsorbent increased with increasing adsorbent dosage. This may be attributed to increased surface area and the availability of more adsorption sites. Using the NaOH-modified adsorbent, the % of Cr removal was very low. This could probably be because NaOH in the modified adsorbent extracts a high amount of hemicelluloses and part of the lignin which are responsible for adsorption in the material being studied. This same effect was observed by Roger (2006).

In the case of Ni, the unmodified adsorbent removed more Ni compared to the HCl-modified and NaOH-modified adsorbents. The percentage of Ni removed increased to 99.77 as the adsorbent loading increased from 0.2 to 0.6 g while a sharp decrease in adsorption was noticed at 0.8 g. This may be due to aggregation of adsorption sites resulting in decrease in total adsorbent

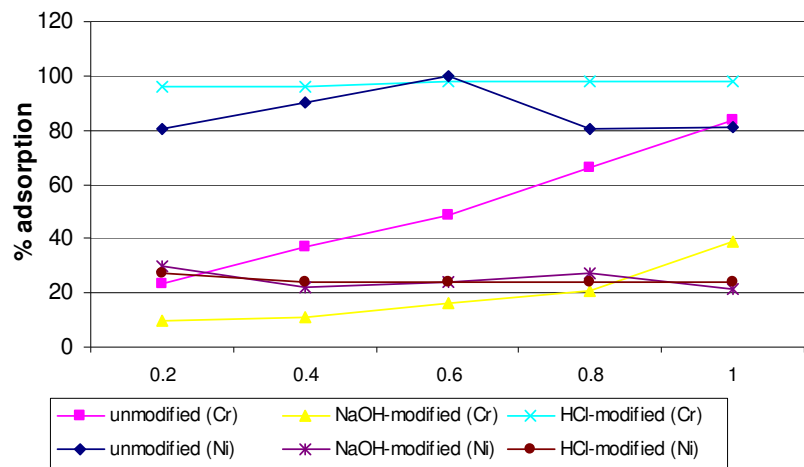


Figure 1. Effect of adsorbent loading on the adsorption of Cr and Ni.

surface area of particles available to Ni and an increase in diffusion path length. The pattern of adsorption for HCl- and NaOH-modified adsorbents are similar.

The effect of concentration on the adsorption of the metal ions

The results of the effect of concentration on adsorption of Cr and Ni are shown in Figure 2. The adsorption of Cr(VI) by HCl-modified adsorbent increased as the initial concentration of the Cr ion was increased. The adsorbent was able to adsorb a concentration of 96 mg/L, when an initial concentration of 100 mg/L solution of Cr(VI) ion was used. This indicated that the binding site was not saturated and could probably still bind more Cr(VI) ion if a higher initial concentration was used. The adsorption of Cr by NaOH-modified and unmodified adsorbents also increased with increase in the initial concentration of the adsorbent, but did not adsorb as much as the HCl-modified adsorbent.

For the removal of Ni, the HCl modified adsorbent removed the least amount of metal ion as the initial concentration of metal ion was increased. The amount of Ni(II) removed was almost constant as the concentration of the initial solution of the synthetic effluent increased from 10 - 100 mg/L. This probably indicated that for an adsorption of a concentration of 8 mg/L of Ni(II) the available binding sites had been saturated and the adsorbent could no longer bind further ions as the number of sites available on the adsorbent is constant. Furthermore, the presence of competing ion (H_3O^+) from the HCl modified adsorbent could also hinder the capability of the Ni ion been adsorbed on the adsorbent.

Effect of pH on the adsorption of metal ions

The adsorption capacity of the adsorbents as a function

of hydrogen ion concentration (pH) was determined. The results for the adsorption of Cr and Ni are shown in Figure 3.

Chromium adsorption was high for all the adsorbents at low pH values of 2, 3, 4 and 5 respectively. The adsorption capacity of all the adsorbents for Cr decreased with increasing pH values, but this was not true for the HCl-modified adsorbent as the values only fell slightly below 80% even at higher pH. Generally, Cr is better adsorbed at low pH values. This could be due to the fact that at low pH, Cr exist as Cr^{3+} and can therefore be better adsorbed by the C=O functional group on the adsorbent since oxygen is electronegative and can attract ions that are positively charged (Sharma and Forster, 1994). Also according to Samanta et al. (2002), at low pH, there is presence of a large number of H^+ ions, which in turn neutralizes the negatively charged adsorbent surface thereby reducing the hindrance to the diffusion of dichromate ions.

In the case of Ni, all the adsorbents had high adsorption of 90 - 99% at pHs of 8 - 9. It is apparent that Ni is strongly adsorbed at higher pH values. The optimum pH for the adsorbents could be between pHs 6 and 8. This could be due to an increasing negative charge density on the adsorbent surface. At pH greater than 8, Ni(II) removal was mostly due to precipitation and not by sorption. A similar result was obtained when sawdust was used as the adsorbent (Olayinka et al., 2007).

Effect of contact time on the adsorption of metal ions

The results for the effect of contact time on adsorption of Cr(VI) and Ni(II) are shown in Figure 4. 20 mg/L of each of the solutions and 0.4 g of the adsorbents were used for this experiment. A pH of 3 was used for Cr solution while pH 7 was used for the Ni solution. It was observed that the HCl-modified adsorbent consistently had a high per-

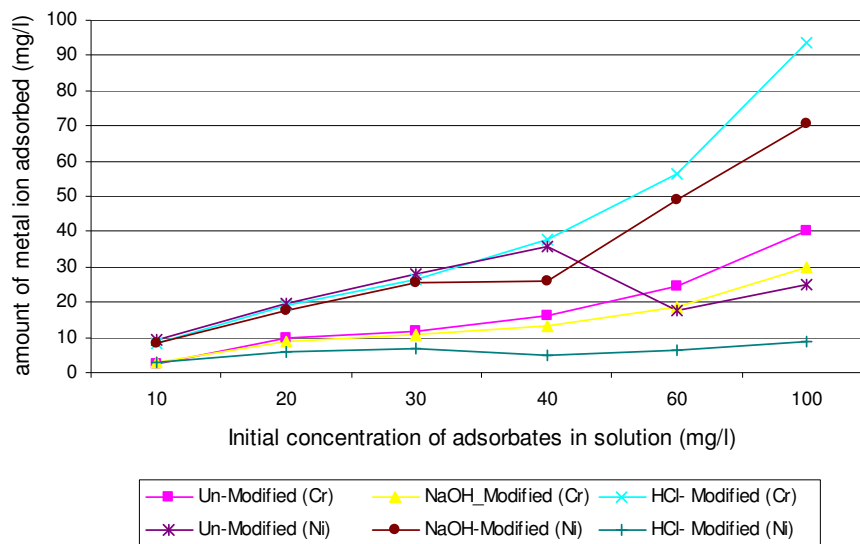


Figure 2. Effect of concentration on the adsorption of ions.

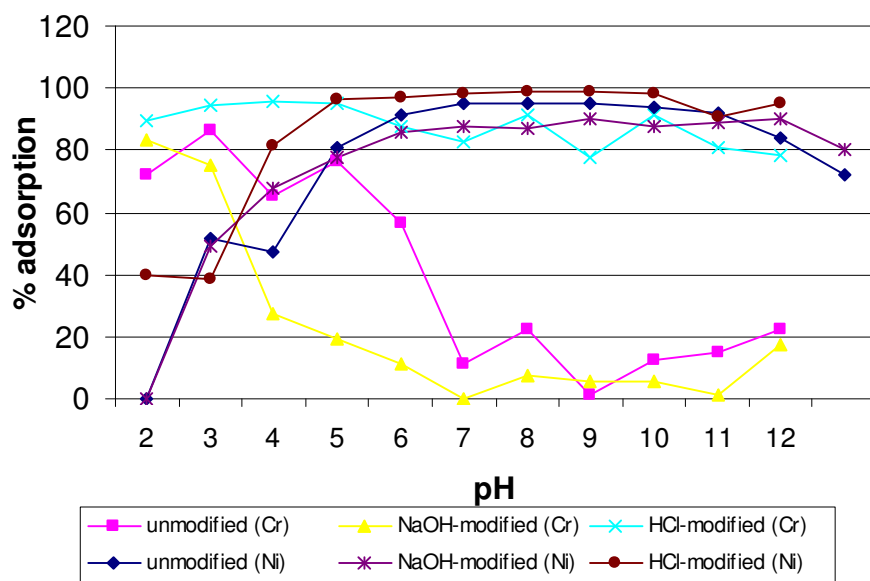


Figure 3. Effect of pH on the adsorption of ions.

centage of adsorption for chromium all through the contact time used which means equilibrium was already reached as early as 30 min. For unmodified adsorbent the percentage of chromium adsorbed increased till 90 minutes and it significantly decreased at 120 min. which could be as a result of desorption of the metal ions.

In the case of Ni, percentage adsorption was consistent. The percentage of Ni adsorbed was constant for each of the adsorbents, the unmodified, NaOH –modified and HCl-modified adsorbents consistently gave adsorption of 95, 88 and almost 50% respectively. It could be said that the metal uptake was rapid and all the metals

adsorbed had been done within 30 min.

Effect of temperature on adsorption of metal ions

The results for the effect of temperature on adsorption of Cr(VI) and Ni(II) are shown Figure 5.

The increase in temperature did not bring about any significant increase in the adsorption capabilities of the adsorbents, rather a decrease in adsorption was observed for all the adsorbents.

The percentage adsorption of Ni (II) by all the adsorbents did not improve, but rather a decrease was observed

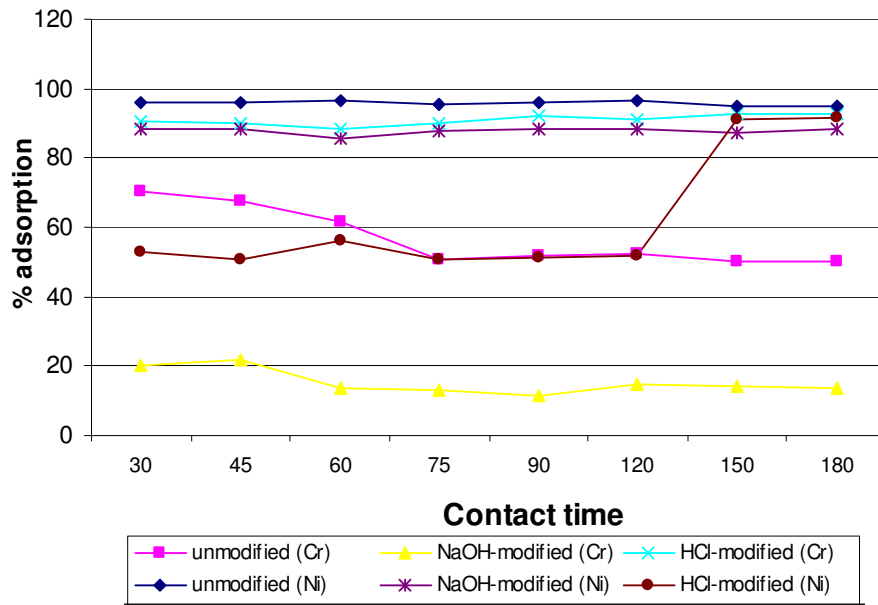


Figure 4. Effect of contact time on the adsorption Cr and Ni.

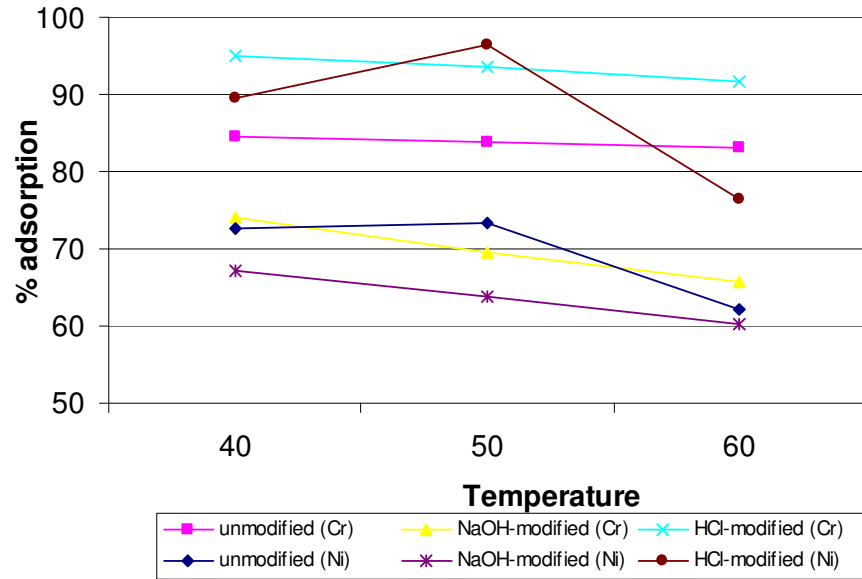


Figure 5. Effect of temperature on the adsorption of Cr and Ni.

in all the adsorbents at temperatures between 50 and 60°C.

Adsorption isotherm study

The isotherm data obtained using both the Freundlich

and the Langmuir adsorption isotherm models are shown in Table 2. The Freundlich adsorption isotherm model was observed to be more suitable for the adsorption of Cr(VI) on coconut husk, while the Langmuir adsorption isotherm model was found to better explain the adsorption of Ni(II) on coconut husk. The graph of the adsorption

Table 2. Adsorption Isotherm equations and regression data for the adsorption of Cr(VI) and Ni(II) on coconut husk.

Adsorbent(adsorbate)	Freundlich adsorption isotherm		Langmuir adsorption isotherm	
	Equation of graph	R ²	Equation of graph	R ²
Unmodified coconut husk (Cr(VI))	$y = 1.1269x - 1.6075$	0.9627	$y = 39.407x - 0.3853$	0.9442
NaOH- Modified coconut husk (Cr(VI))	$y = 0.9501x - 1.3949$	0.9595	$y = 33.904x - 0.1601$	0.9573
HCl-Modified coconut husk (Cr(VI))	$y = 1.0451x - 0.1112$	0.9978	$y = 12.269x - 0.0351$	0.9954
Unmodified coconut husk (Ni(II))	$y = 0.3201x + 0.211$	0.4373	$y = 6.7751x + 0.2921$	0.7673
NaOH-Modified coconut husk (Ni(II))	$y = 0.9193x - 0.9641$	0.9961	$y = 11.438x + 0.0136$	0.9987
HCl-Modified coconut husk (Ni(II))	$y = 0.4005x - 0.8383$	0.7767	$y = 23.713x + 0.8474$	0.9173

Table 3. Kinetic equation and regression data for the adsorption of Cr(VI) and Ni(II) on coconut husk.

Adsorbent (adsorbate)	Equation of graph	R ²
Unmodified coconut husk (Cr(VI))	$y = 0.1974x + 6.2638$	0.7634
NaOH- Modified coconut husk (Cr(VI))	$y = 3.0826x - 102.99$	0.7411
HCl-Modified coconut husk (Cr(VI))	$y = 0.1187x + 0.1206$	0.9997
Unmodified coconut husk (Ni(II))	$y = 0.105x + 0.0188$	0.999
NaOH-Modified coconut husk (Ni(II))	$y = 0.1156x - 0.1872$	0.9998
HCl-Modified coconut husk (Ni(II))	$y = 0.1936x - 0.1122$	0.9995

of Cr(VI) on coconut husk using the Freundlich isotherm model had a high value for regression correlation coefficient (R²) which ranged from 0.9595 - 0.9978, with the HCl-modified adsorbent having the highest R² value. The relatively higher R² value of the graph of Ni(II) adsorption using the Langmuir Isotherm model as compared to the Freundlich isotherm model confirmed a monolayer adsorption of Ni(II) on the coconut husk surface.

Adsorption kinetics

To determine the kinetics of adsorption of Cr(VI) and Ni(II) using coconut husk, pseudo-first order and second order models were tested on the experimental data.

The regression correlation coefficient obtained from the pseudo-first order kinetic graph was observed to be quite low and this implied the non-applicability of the pseudo first order kinetic model to the experimental data of the adsorption of both Cr(VI) and Ni(II) on coconut husk.

Application of second-order kinetic model kinetics by plotting t/N_t vs t gave high values of regression correlation coefficient for both Cr(VI) and Ni(II) adsorption on coconut husk (Table 3). This implied that the mechanism of adsorption of both ions on coconut husk followed the second order kinetics.

Conclusion

For the optimization of the different parameters varied, the adsorbents gave different results. HCl modified adsorbent gave the best result of all the adsorbents as about 96% adsorption was attained at pH 5 with 0.4 g of the adsorbent, 20 mg/L of metal concentration and a contact time of 90 min for the adsorption of Cr(VI). This is an improvement over the 84% reported by Rao et al. (2002) and 99% reported by Olayinka et al. (2007) as the mass of adsorbents used by them ranged between 1 and 5 g. The result of this present study perfectly agrees with that reported by Garg et al. (2004) on the use of treated sawdust in the adsorption of Cr(VI) as the same mass of the adsorbent gave a similar result.

The removal efficiency of unmodified adsorbent was found to be far greater than that observed for NaOH-modified adsorbent in the removal of Cr, even though was less than that of HCl-modified adsorbent. The modified adsorbent was found to be the poorest in adsorption as clearly shown in all the Figures. This is because sodium hydroxide can extract major amount of the hemicelluloses and part of the lignin which are responsible for adsorption in the materials been studied.

The Freundlich adsorption isotherm model was observed to be better suited for the adsorption of Cr(VI) on

coconut husk (HCl-modified, unmodified and NaOH-modified), with the HCl-modified having the best R^2 value, while the Langmuir isotherm model was found to be better for the adsorption of Ni(II) on coconut husk. The mechanism of adsorption of the ions investigated can be explained using the second-order kinetics.

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