

EFFECTS OF PROCESS PARAMETERS ON THE ADSORPTION OF HEAVY METAL IONS BY CACTUS ASH RESIDUE

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ABSTRACT

Heavy metals contamination exists as aqueous waste polluting water sources causing health challenges. The objective of this study therefore is to determine the adsorption parameters heavy metals removal from water. Batch experiment was conducted using cactus ash residue as adsorbent and pH ranges of 2 to 11, at 25 to 60°C. The result shows optimum removal dosage of Cr and Ni to be 0.4g dose of cactus ash and 0.5g for Pb and Cd. Increase in pH was found to increase the adsorption of the aforementioned heavy metals. An optimum pH for the adsorption of the heavy metals was found to be pH of 7, which implies that neutral pH condition is more suitable for optimum uptake of the heavy metals. The adsorption process occurred rapidly when cactus ash residue adsorbent was used with almost 96 to 99% Cr, Ni, Pd and Cd present adsorbed after 10 minutes. It was observed that the adsorption capacity decreased with increased in temperature from 25 to 60°C. Adsorption of the Cr, Ni, Pb and Cd was not affected by increase in temperature between 25 to 30°C. The adsorption of heavy metals from water sample was studied and found to be greatly influence or affected by the aforementioned process parameters. Cactus ash residue can be effectively used for the treatment of heavy metals polluted effluents.

Keywords: Adsorption, heavy metals, water, cactus, pollutants

1 INTRODUCTION

Heavy metal pollution has become one of the most serious environmental problems today. Unlike organic pollutants, heavy metals are non-biodegradable and they can be accumulated in living tissues, causing various diseases and malfunction in human [1]. The presence of toxic heavy metals in the environment is of great concern due to their persistence in nature and chronic adverse effects on human health and the environment due to their presence in many environments as pollutants in many on land, air and water bodies [2]. Industrial development achieved over the years has been one of the major causes of the rise in environmental challenges affecting the natural setting of our ecosystem. For instance, water an important part of the ecosystem used by man for consumption, farming, hygiene, industry etc., has been contaminated by human activities leading to the disposal of waste in the environment. The natural state of water has been compromised by the indiscriminate discharge of industrial waste in water bodies. Heavy metals and dyes are two examples of the largely widespread pollutants in industrial effluents that may have severe problems for the environment including the human health [3]. Several studies have been conducted on how to restore the sanity of our environment which is littered with heavy metals. Some of the conventional method for the remediation of our water bodies include activated sludge, coagulation-flocculation, chemical precipitation, adsorption and filtration while other established methods include: ion exchange, oxidation, solvent extraction, electrochemical treatments, membrane bioreactors, etc. Emerging

technologies used for the removal of heavy include advanced oxidation, biosorption, adsorption into non-conventional materials, nanofiltration, etc [4]. Adsorption, which is considered safe, clean, efficient and technical feasible process, is frequently employed to remove various pollutants such as heavy metals and dyes making adsorption method an interesting separation technique by using suitable and local materials such as plant leaves, saw dust and other lignocellulosic material for developing adsorbent characterized by high surface area and porosity and allowing rapid adsorption equilibrium kinetics [5, 6]. Adsorption is one of the most common methods used in wastewater treatment because of it is economical, effective and simple in design. However, the adsorption process is influenced by the several parameters such as pH, temperature, contact time, adsorbent dose etc. Adsorption is generally considered to be a spontaneous process with an exothermic nature, and an increased uptake of organic molecules is expected when the adsorption temperature decreases. One of the major advantages of adsorption is the use of low-cost adsorbents like agricultural biomasses such as fruit peels, fruit shells, agricultural waste, plant leaves etc. [7]. As stated by Al-senani and Al-fawzan [8] there are many methods of adsorption of heavy metals from the environment, both chemical and physical with most of the methods not economically feasible, hence the need for exploring low-cost effective alternatives such as Cactus ash residue. Several parameters such as temperature, pH, contact time etc. influence the optimum performance of adsorbent in heavy metal removal from water. The effect of such factors varies with the type of material used as adsorbent. The influence of the aforementioned parameters for Cactus ash adsorbent has not been studied, hence the aim of this study. The knowledge of the influence of such parameters will aid in reactor design with a condition for optimum Cactus Ash adsorbent performance for heavy metal removal from water. Cactus is a member of the plant family called Cactaceae which originated from arid and semi-arid zones. The Cactaceae family consists of large number of genera and species, which were found in many regions over the world (South America, North Africa, Australia, Asia, etc.) [3]. The plant contains protein and lipid at low concentrations, however carbohydrates represent the major component for all samples (percentage on dry weight base ranged between 40 and 93%) [9]. As stated earlier, adsorption process is influenced by several factors, which have significant influence on the sorption capacity of the sorbent or sorption rate. Thus, need to be taken into account [8]. The most common one includes the nature and dose of adsorbents, nature and concentrations of adsorbate, temperature, modifying reagents (in case of natural adsorbent), contact time, Interfering cations, (co-ions effect) speed of agitation particle size and pH of the aqueous solution [9]. Solution temperature plays a significant role on the adsorption by influencing the equilibrium and rate of adsorption processes but generally, increased adsorption capacity with increasing temperature is known as an endothermic process and decreased adsorption capacity with the increase in solution temperature is termed as exothermic in nature [10]. As stated by Bouguettoucha *et al.* [11] who studied the adsorption of heavy metal ion and dye from water by agricultural waste solid adsorbent, increase in temperature often rises the mobility of adsorbate (heavy metals or dyes) molecules with an increase in their kinetic energy, as well as an enhanced rate of intraparticle diffusion of sorbate. Cermakova *et al.* [12] who studied the influence of temperature on the adsorption using activated carbon reported that, higher temperature increases the diffusion of the adsorbent, reduces the solution viscosity and enhances hydrophobic interactions leading to adsorption. Study conducted by [13] on the removal of Cu, Pb and Cd shows high removal rate of Cu up to pH of 6 while Pb and Cu present high adsorption rate above pH of 5. Studies shows that increase in pH affects the removal efficiency for Cd, Pb, and Cu while on the other hand, the removal efficiency for Pb, Cu, and Cd was 20%e45% at low pH values (pH 2.0e3.0) [13]. The biosorption of Pb (II), Zn (II), Cu (II) and Cd (II) onto activated carbon prepared from live branches was found to be pH dependent with a maximum metal ion biosorption achieved at pH of 5 for Pb, Cu, Cd ions and pH of 3 for Zn ions. Also, metal biosorption is exothermic in character and increases with temperature [14]. Several studies were carried out using different adsorbent but none has been reported on adsorbent made from cactus ash for the adsorption of heavy metals from water. This study therefore explores the effect of process parameters on the performance of cactus ash adsorbent which can be developed easier and cheaper for water treatment.

2 METHODOLOGY

2.1 Sample Collection and Pretreatment

The cactus plant was collected from the environs of Girei Local Government Area, Adamawa State, washed and identified by plant botanist in the Department of Botany, Modibbo Adama University Yola. The plant was washed with distilled water to remove dirt, according to the method adopted from [1].

2.2 Apparatus and Equipment/Work Materials

- i. Cactus plant
- ii. Measuring Cylinders
- iii. Volumetric Flasks
- iv. Beakers and Conical Flask
- v. Spatula
- vi. Stirrer
- vii. Whattmann Filter Paper
- viii. Funnels
- ix. Wash Bottles/Sample Bottles

2.3 Preparation of Cactus Ash Residue

The clean plant sample was cut into strips of 1 to 2 cm, dried in an oven at 60° C for 24 hours. Ash was obtained as dry plant material was heated in a furnace at 600 °C with excess air for 24 hours. Ash residue was prepared by dissolving the ash in distilled water to remove the soluble alkali, filtered, washed with distilled water and dried in an oven at 100°C [1].

2.4 Reagents and Equipment

The type of reagents and equipment of machine that was used for this study is outlined in Table 1 and 2 respectively.

Table 1. List of Reagent Used

Reagent	Purity/Model	Manufacturer
Hydrogen Chloride	35-38%	BDH Limited poole England
Sodium Hydro Oxide	96.0%	Central Drug House P Ltd India
Potassium Chromate	99.0%	Labtech Chemicals
Lead Acetate	99.0%	Labtech Chemicals
Cadmium (metal)	99.0%	Lobal Chemie
Nickel Chloride	99.0%	Lobal Chemie

Table 2. Equipment/Instruments and Model Used for This Research Work

Equipment	Model	Manufacturer
FT-IR Spectrophotometer	version 10.03.06	Perkin Elmer
Microwave Oven	OV/100	Gen Lab.
Electronic Weighing Balance	PW184	ADAM Equip.
Orbital Shaker	SSL1	Stuart
Incubator Shaker	S1500	Stuart
Atomic Absorption Spectrophotometer	205	Buck Scientific
Digital pH Meter	PH209	HANNA INST.
XRF	EDXRF	Rigaku Nex De
Muffle Furnaces	EC F2	Vecstar Ltd

2.5 Preparation of Metal ion Solutions

Analytical grade compound containing metal element were obtained from a Biochemistry laboratory, Gombe State University. A stock solution of metal ion of concentration 1000 mg/L was prepared by using molar mass of compound containing metal element divide by the atomic mass of the corresponding metal element by dissolving the required gram of the chemical in 1 L of distilled water. An experimental metal ion solution of desired concentration was obtained by appropriate dilution of the stock solution.

2.6 Fourier transform infra-red (FTIR) spectroscopy

Fourier transform infra-red (FTIR) spectrophotometer was used to identify the different functional groups available on the adsorbent sites. The FTIR of the adsorbent was taken before adsorption using FTIR spectrophotometer. 0.1 g of cactus ash residue as adsorbent was encapsulated with 1 g of KBr spectroscopy grade and by introducing the mix in a piston's cell of a hydraulic pump with compression pressure 15 KPa/cm²; the solid translucent was obtained which introduced in an oven for 4hrs at 105°C which ensured the non-interference of any existing water vapors or CO₂ molecules. The FTIR spectrum was then recorded within the wave number range 4000 – 450 cm⁻¹

2.7 XRF Analysis

Sample was first grounded into a homogenous powder approximately 100-200 mesh, and then about 5g of the sample was weighed into 32 mm sample cup with a polypropylene X-ray film of 4µm thickness and was hydraulically pressed. Sample heights was measured in millimetres and sample cup was capped. The EDXRF analysis was performed using a Rigaku NEX DE EDXRF spectrometer equipped with a fifteen-place sample changer with spin function using slow and steady spinning mode. EDXRF measurement was carried out under helium (He) atmosphere using a palladium (Pd) X-ray tube at a voltage of 60 kV and current 10µA with 10 mm beam spot size, and silicon (Si) drift detector comprised of Peltier electronic circuit cooling system. Standard less calibration was employed using Fundamental Parameters (FP) methods software (Rigaku RPF-SQX) to allow for elemental quantification of completely unknown. The software automatically estimated elemental concentration and provided appropriate corrections. Rigaku NEX DE EDXRF spectrometric analysis was employed to measure a wide range of atomic elements, from sodium (11) through uranium (92), while providing elemental detection limits from low parts-per-million (ppm) to high weight percent (%wt) The analytical uncertainties was generally within 0.1 to 1% (RSD).

2.8 Batch Adsorption Experiments/ Parameters

Batch tests were carried out in 120ml plastic bottles to check the influence of various parameters such as pH, adsorbent concentration (dose), contact time and temperature. The experiments were carried out at the same shaking speed. For each experimental run, 50 ml aqueous solution of known concentrations of Cr, Ni, Pb and Cd ions were put in 120ml plastic bottles that contained known amount of cactus ash residue a combusted product. These bottles were shaken at a constant shaking rate of 150 rpm and at room temperature and filtered. At the end of each experiment, plastic bottles were removed from the shaker and solutions separated from the adsorbents by filtration through filter paper. The concentrations of Cr, Ni, Pb and Cd ions in the filtrates obtained were measured using flame atomic absorption spectrophotometer. The amounts of Cr, Ni, Pb and Cd adsorbed per unit mass was calculated. The pH of the medium was adjusted using HCl and NaOH solution.

2.8.1 Effect of adsorbent dose

Adsorbent dosage was varied from 0.1 g to 0.5 g so as to determine the optimum adsorbent dosage for the removal of Cr, Ni, Pb and Cd ions. The experiment was carried out in triplicate at room temperature, pH of 7.0 and concentration of 100 mg/L for the ions. Samples were ten shaken at constant oscillation of 150 rpm for 2 hours, filter and analyze using AAS for the metal ions adsorption.

2.8.2 Effect of initial concentration

The effect of initial concentration on adsorption of metal ions was investigated on 0.5g/50ml of the adsorbent while varying concentration of metal ions in the range 20 to 100 mg/L. The mixtures in triplicate were shaken at a speed of 150 rpm at room temperature and pH 7. The samples were then filtered and the resulting filtrate analyzed for the remaining metal ions using AAS. Average values were used to calculate the amount and percentage of metal ions concentrations.

2.8.3 Effect of temperature

Adsorption experiments were carried out by varying temperatures at 25, 35, 45, 55 and 65°C in an incubator shaker and maintaining a dosage of 0.5g of the adsorbent, contact time of 2 hours, shaking speed of 150 rpm, pH of 7, and concentration of 100 mg/L Cr, Ni, Pb and Cd. The resulting mixtures were collected, filtered and analyzed.

2.8.4 Effect of pH

Adsorption experiment at different pH of 2 to 12 was carried out at a room temperature and concentrations of 100 mg/L. The pH of the solutions was adjusted by adding HCl, NaOH drop wise using teat pipette. Adsorbent of 0.1 g were weighed accurately and then added to 50 ml solution portions of Cr, Ni, Pb, and Cd at pH 2, 5, 7, 9 and 12 and mixture agitated for 2 hours. The resulting mixture was filtered and the concentrations of the ions water was analyzed.

2.8.5 Effect of contact time

50 ml of 100mg/L Cr, Ni, Pb and Cd of the adsorbate was transferred in stoppered plastic bottles and agitated with 0.5g of the adsorbent. Agitation was carried out at constant oscillation of 150 rpm for 10, 30, 60, 90 and 120 minutes respectively, and samples were filtered and analyzed.

3 RESULTS AND DISCUSSION

3.1 Fourier Transform Infra-Red (FTIR) Spectroscopy

The FTIR spectra of adsorbents were used to investigate the functional groups present on adsorbent that could be responsible for the removal of heavy metal ions. The spectra of the cactus ash residue were measured within the spectral range of 4000 – 450 /cm as shown in Figure 1. Various adsorption bands were observed such as prominent peak at 3694.66 cm^{-1} shows the trapped CO₂ band peak intensity is 3-fold greater than for the strongest. The peak at the region of 3423.23 cm^{-1} due to hydroxyl group, O-H bond, probably attributed to adsorbed water. The peak at 2923.58 cm^{-1} was attributed to C-H interaction with the surface of the adsorbent indicating the carbon dioxide of normal air. The peak at 1434.93 cm^{-1} shows the presence of C=H, C=O inorganic carbonate. Peak at 1048.25 cm^{-1} shows the presence of C-O bond confirming presence of carboxylic acid. The band at 873.68 cm^{-1} bending -C=O inorganic carbonate. The peak at 712.89 cm^{-1} shows CH out of plane aromatic band [13].

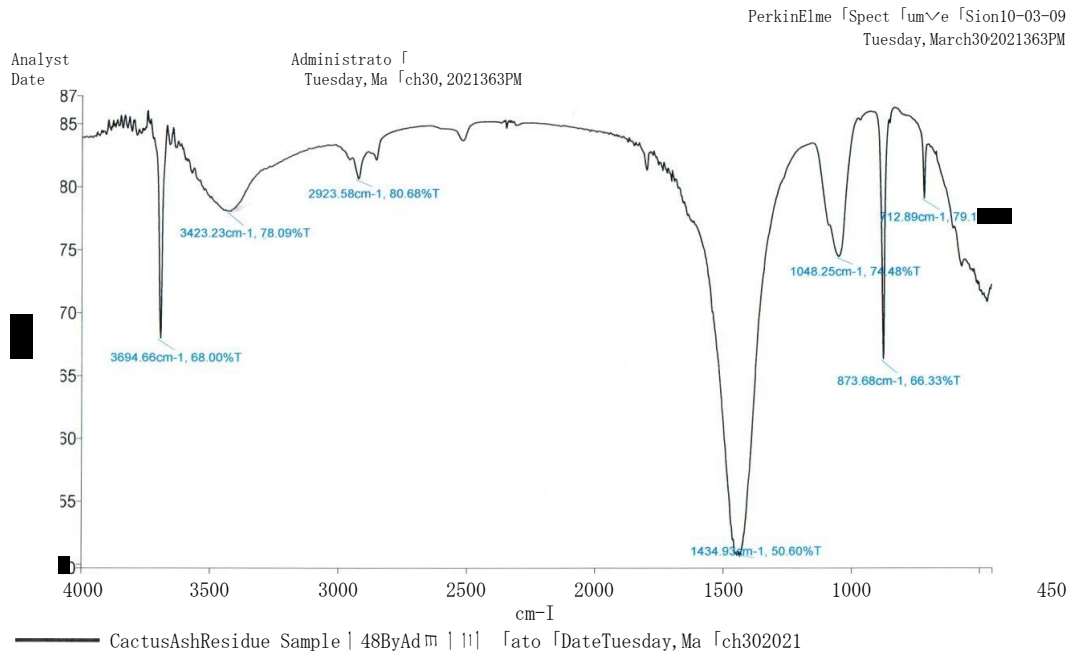


Figure 1. Fourier Transform Infra-Red (FTIR) Spectroscopy

3.2 Batch Adsorption Experiments/ Parameters

3.2.1 Effect of adsorbent dose

The percentages removal of Ni and Cr ions increased with the increasing of adsorbent doses, it's also the same for the case of Pb and Cd. At the first 0.1g dose cactus ash residue, the heavy metal removal capability of cactus powder increased significantly for both Ni, Cr, Pb and Cd ions from 96.93 to 98.79% for the optimum dose of 0.4g and it goes down to 98.77% for 0.5g dose for Cr ion, from 98.24 to 99.68% at the dose of 0.4g as optimum dose also and slightly goes down for the 0.5g dose to 99.64 for Ni ion, from 92.59 to 97.60% for Pb ion and from 95.87 to 98.16%, for 0.1 to 0.5g respectively. Once the water sample is activated, the percentage removal of each ion by cactus powder kept increasing up to 0.4g of cactus powder. After 0.4g, at 0.5g of cactus powder, the removal efficiency decreased rapidly to 98.77%. This indicates that to remove Cr metal from water sample a minimum 0.4g of cactus powder is required. In similar way, when the cactus ash residue increased from 0.1 to 0.4g, the percentage removal of Ni ion increased from 98.24 to 99.68%, and slight decrement occurs at 0.5g of cactus powder. Further increasing of the quantity of cactus powder did not show any significant change on the removal of Ni from water sample. This is due to the fact that active site of the cactus powder already occupied by Ni ion, and the solution is reaches equilibrium between the Ni ion and the cactus powder, so also for Pb ion and Cd the optimum conditions are both on the dosage of 0.5g. The observed enhancement in adsorption yield with increasing adsorbent dosage could be due to an increase in the number of possible binding sites and surface area of the adsorbent [12]. The effect of the adsorbent dose was investigated by retaining different doses of cactus ash residue and the data obtained is shown in Figure 2.

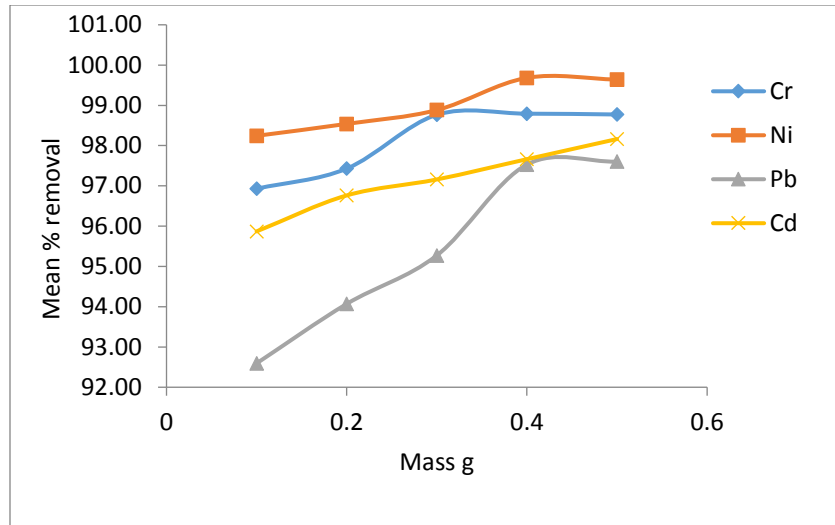


Figure 2. Effect of adsorbent dosage on percentage removal of Cr, Ni, Pb and Cd

3.2.2 Effect of PH

The pH of the aqueous solution is one of the major parameters controlling the adsorption process. To demonstrate the effect of pH on adsorption uptake of Cr, Ni, Pb and Cd ions on the adsorbent as a function of pH, adsorption was studied in the pH of 2, 4, 7, 9 and 11, and the data obtained are given in Table 2, and Figure 3. The adsorption capacity of cactus ash residue increased from 90.32 to 98.81% for Cr, 95.11 to 99.70% for Ni, 91.53 to 97.56% for Pb and 93.37 to 98.18% for Cd ions when the solution pH was increased from 2 to pH 7. At lower pH values, the adsorption of ions is significant. Subsequently the surface becomes negatively charged and this increases the adsorption of the positively charged ions through electrostatic forces of attraction, and the solution pH 7 shows the optimum adsorption. The decrease in adsorption of the ions for pH greater than 7 is due to the complexation of metal ions by OH^- groups which would prevent the metal adsorption [14].

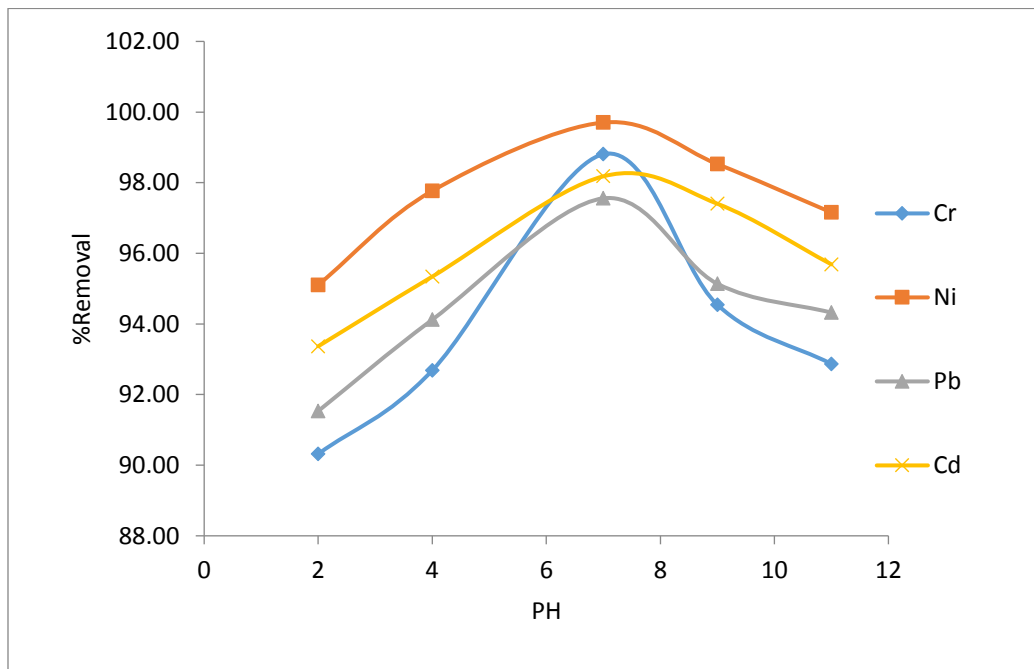


Figure 3. Effect of pH on percentage removal of Cr, Ni, Pd and Cd

3.2.3 Effect of Contact Time

The effect of contact time on the amount of Cr, Ni, Pb and Cd ions adsorbed was investigated at initial concentration of 20 mg/L of the metal ions. The result shown in Figure 4, the adsorption process occurred rapidly when cactus ash residue adsorbent was used with almost 96 to 99% Cr, Ni, Pd and Cd present adsorbed after 10 minutes. Between 10 minutes and 120 minutes there was no significant difference there in percentage removal. The equilibrium was fully reached after contact time of two hours with the difference between the adsorptive uptake at 90 minutes and 120 minutes for all the metal ions being less than 1.0%. Hence, a steady-state approximation was assumed and a quasi-equilibrium situation was considered at $t = 2$ hrs [10]. The results relate well with previous studies [14].

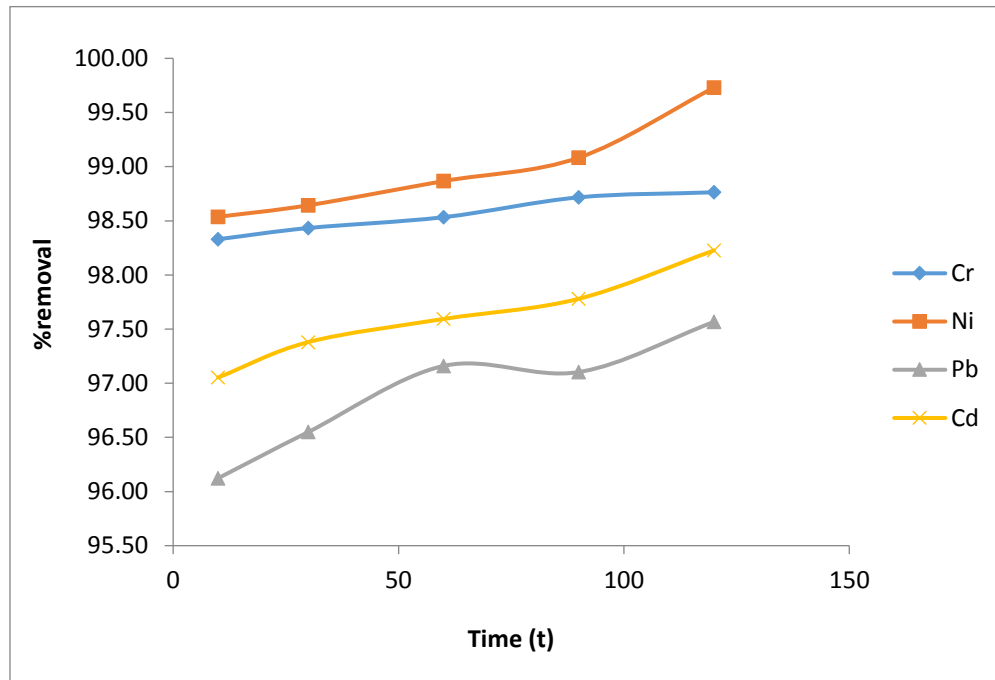


Figure 4. Effect of time on the percentage removal of Cr, Ni, Pb and Cd

3.2.4 Effect of temperature

Experiments were carried out at temperatures of 25, 30, 40, 50 and 60 °C. It was observed that the adsorption capacity of cactus ash residue decreased with increased in temperature from 25 to 60°C, which indicates a low temperature favors adsorption of the metal ions. This shows that surface activity decreases at higher temperatures. The decrease in adsorption capacity of Cr, Ni, Pb and Cd at high temperature shows weak interaction between the active sites and metal ions supporting physio-sorption where the reaction is exothermic and favored by low temperature. Figure 4.6 shows that the adsorption of Cr and Pb onto cactus ash residue was not affected by temperature increase from 25 to 30°C, likewise Ni and Cd at 25°C to 40°C. This suggest the process was endothermic. Similar results were reported for Cd and Pb removal using rice as biosorbent. The effect of temperature on adsorption capacity of Cr, Ni, Pb and Cd by cactus ash residue was investigated as a function of temperature and data obtained summarized in Figure 5.

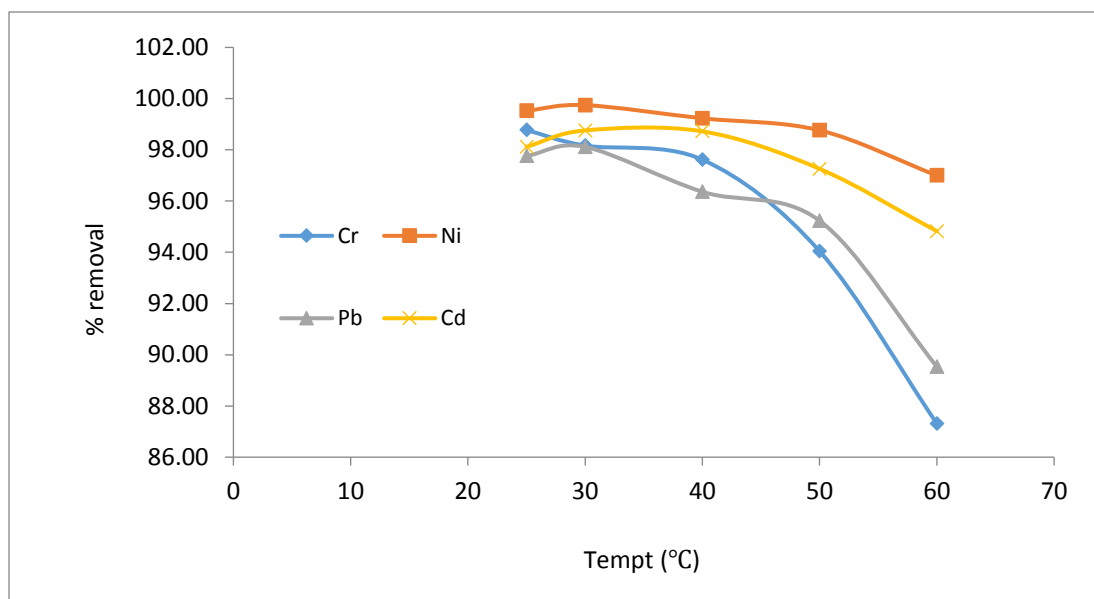


Figure 5. Effect of temperature on percentage removal of Cr, Ni, Pb, Cd

4 CONCLUSIONS

The adsorption Chromium, Nickel, Lead and Cadmium ions on Cactus Ash Adsorbent was studied in a batch system. The adsorption process was found to be influenced significantly by the process parameters. Optimum removal dosage of Cr and Ni was found to be 0.4g dose of Cactus Ash and 0.5g for Pb and Cd. Adsorption uptake of the heavy metals was studied in the pH ranges of 2 to 11. An optimum pH for the adsorption of the heavy metals was found to be pH of 7, which implies that neutral pH condition is more suitable for optimum uptake of the heavy metals. Increase in pH was found to influence the uptake of Cr ion the most with an uptake rate of 90.32 to 98.81%. The adsorption process occurred rapidly when cactus ash residue adsorbent was used with almost 96 to 99 % Cr, Ni, Pd and Cd present adsorbed after 10 minutes. Between 10 minutes and 120 minutes there was no significant difference in percentage removal. Adsorption capacity and temperature were found to have an inverse relationship. Low temperature favors higher adsorption of the metals. Adsorption of the Cr, Ni, Pb and Cd was not affected by increase in temperature between 25 to 30°C.

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