CHAPTER 1

INTRODUCTION

1.1 Problem Statement

The waste water effluent from ethanoic acid producing plant, BP-PETRONAS Acetyls Sdn Bhd, contains metal ions which are Ni^{2+}, Cr^{3+}, and Fe^{3+}. These metal ions need to be removed from the waste water before the water being channeled to the environment, conforming to the legal requirement. The heavy metals are very toxic in nature and can cause harm to the environment.

1.2 Objective

There are few objectives to be achieved in this research project as below:

- To simultaneously remove the three metal ions (Ni^{2+}, Cr^{3+} and Fe^{3+}) from the waste water the BPPA plant using mixed adsorbents.
- To obtain the minimum amount and correct ratio of Amberlyst 15 (Wet) resins, and activated carbon, to produce an adsorbent mixture that can produce maximum removal of Ni^{2+}, Cr^{3+} and Fe^{3+} ions from the waste water.
- To reduce the waste water treatment costs of the plant because currently the waste water are sent to Kualiti Alam Sdn Bhd for treatment and disposal.

1.3 Scope of Study.

The main scope of the study done by the writer is Ni^{2+}, Cr^{3+}, and Fe^{3+} removal from industrial waste water through adsorption process using ion exchange resins and activated carbon. In this study, the performance of pure ion exchange resins, pure activated carbon, and mixture of ion exchange resins and activated carbon will be compared, to identify the best method of the metal ions removal.

1.4 Relevancy of The Project

This study is conducted using effluents taken from BP PETRONAS Acetyls Plant which the outcomes of this project may give new alternative for effluent treatment on-site not only for BPPA plant but to other industries as well. This is to ensure the quality of the environment is maintained and at the same time may help in reducing cost of having to treat the effluent off site.

1.5 Feasibility

This project can be done within the time frame since the chemicals and equipments needed are readily available in the laboratory. Therefore, the scope of study can be accomplished.

CHAPTER 2

LITERATURE REVIEW

2.1 Adsorption

Adsorption is defined as the process when one or more component in gaseous or liquid stream, which is called the adsorbate, is adsorbed on the surface of a solid adsorbent. Adsorption, ion exchange, and chromatography are sorption processes in which certain adsorbates are selectively transferred from the fluid phase to the surface of insoluble, rigid particles suspended in a vessel or packed in a column The adsorbent is usually particle which is arranged forming a fixed bed. The adsorbate can be recovered and the adsorbent which is solids, will be ready to be used for the next adsorption process. In the industry such as in waste water treatment, the adsorption process is implemented in large scale by either a batch, or continuous flow system.

2.1.1 Adsorption Process

When the fluid containing the solute flows past the particles, the solute diffuses from the bulk fluid to the exterior surface of the particle. The solute then diffuses inside the pore to the surface of the pores of the particles. Finally the solute is adsorbed on the surface.

Depending on the types of forces between the fluid molecules and the molecules of the solid, adsorption may be classified as physical adsorption or also known as Van Der Waals adsorption or chemisorption also known as activated adsorption.

2.1.2 Properties of adsorbents:

The adsorbents in an adsorption process have few properties. The adsorbents are highly porous. The adsorption often occurs as a monolayer on the surface of the fine pores of the adsorbents. Usually, Van der Waals adsorption occurs between the adsorbed molecules. The adsorbents have high abrasion resistance, high thermal stability and small pore diameters. The small pores results in higher exposed surface area. This increases the surface capacity for the adsorption process to occur. The adsorbate stream need not to be cooled before flowing it through the adsorbents, as the adsorbents have high thermal stability.

To be suitable for commercial applications, a sorbent should have:

- high selectivity to enable sharp separation
- high capacity to minimize the amount of sorbent needed
- favorable kinetic and transport properties for rapid sorption
- chemical and thermal stability
- extremely low solubility in the contacting fluid
- hardness and mechanical strength to prevent crushing and erosion
- free flowing tendency for ease of filling or emptying vessel
- high resistance to fouling for long life
- no tendency to promote undesirable chemical reactions
- capability of being regenerated
- relatively low costs
-

2.1.3 Adsorption Isotherms:

Adsorption isotherms are the equilibrium between the concentration of solute in the fluid phase and the concentration on the solid at a given temperature. The equilibrium is usually expressed in terms of:

- concentrations (if the fluid is liquid)
- partial pressure (if the fluid is gas

- solute loading on the adsorbent expressed as mass, moles, or volumes of adsorbate per unit mass.

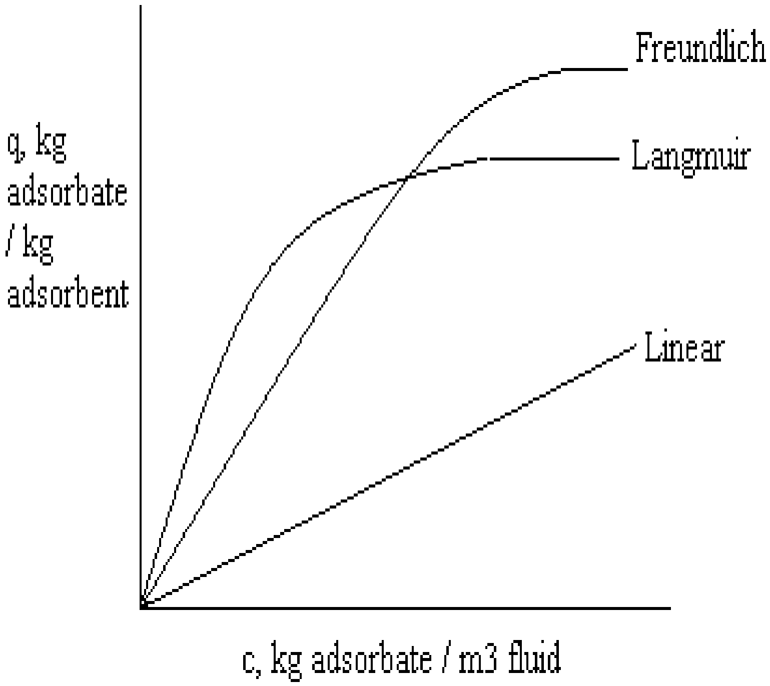


Figure 2.1: Adsorption Isotherms

The convex upwards isotherm as shown in figure 2.1 are favorable because high solid loading can be achieved at a low concentration in the fluid. The linear law of the isotherm is expressed as:

$q = Kc$,
K = constant determined experimentally, m3/kg adsorbent
q = weight adsorbed per unit weight of adsorbent

Freundlich law is expressed as:

$$\mathbf{q = K\ C^n}$$

K and n are coefficients and determined experimentally

q = weight adsorbed per unit weight of adsorbent

C = concentration in fluid

Taking logs and rearranging:

$$\mathbf{\log q = \log K_f + n \log C}$$

Langmuir Isotherm is given by the relation

$$\mathbf{q = q_o\ c\ / (K + c)}$$

q_o = adsorbate loading (kg adsorbate/kg solid)

c = conc in fluid

K = adsorption constant (determined experimentally, kg/m3 adsorbent)

Langmuir isotherm is derived assuming there are fixed number of active sites available for adsorption. Hence, this relation works well only for gases that are weakly adsorbed. Freundlich Isotherm provides a better fir for the adsorption of adsorbate from the liquid phase.

The typical types of isotherms are shown in the figure 2.2. An isotherm which concave upward is unfavorable because low solid loadings are obtained.

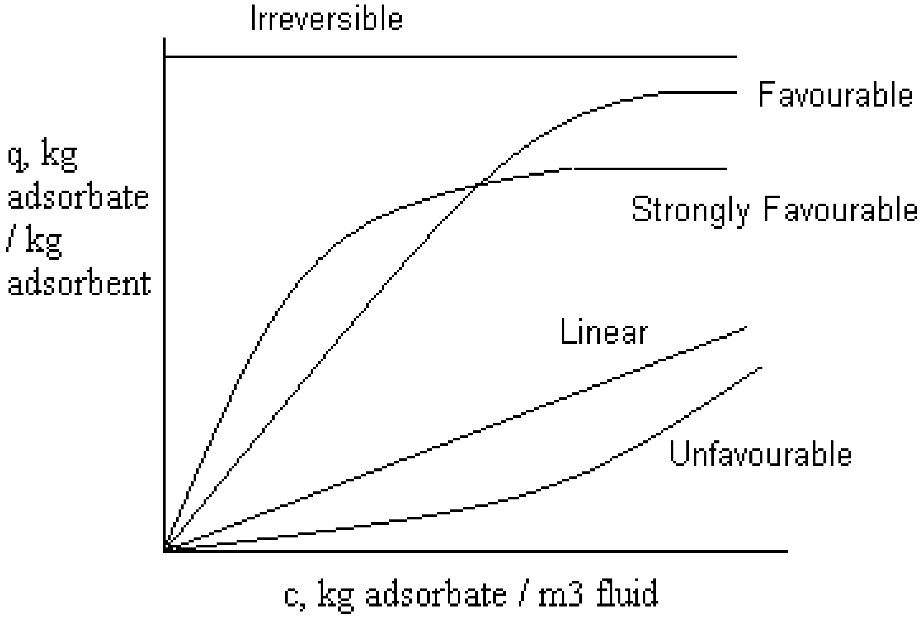


Figure 2.2: Typical types of adsorption isotherms

Adsorbate can be removed from the by raising the temperature. However desorption requires high temperature when the adsorption process is strongly favorable or irreversible than when the isotherm is linear.

2.2 Ion-Exchange Process

Ion exchange is a process when solid particles containing exchangeable cations or anions contacted with electrolyte solution to change the composition of the solution. There are two types of ion exchangers which is anionic and cationic ion exchangers. The cation exchangers includes strong-acid resins with sulfonic acid groups ($-SO^{3-}$) and weak acid resins carboxylic acid group ($-COO^{-}$) and balanced by mobile counterions such as H^{+}, or Na^{+}.

The anion exchangers have strong base quaternary ammonium group $[-N^{+}(CH_3)_3]$ or weak base amine groups ($-N^{+}H_3$) and balanced by mobile counter ions such as Cl^{-}, or OH^{-}. Ion exchange process occurs when the activity of ions in the external solution differs from that of the mobile ions in the resin phase. Example of ion exchange process are shown as below:

Na^+(solution) + HR(solid) $\longleftrightarrow$ NaR(solid) + H^+ (solution)
Cl^- (solution) + RNH3OH(solid) $\longleftrightarrow$ RNH3Cl + OH^-

The ion exchange resins are insoluble in water. The capacity of ion exchanger is number of changeable groups per unit mass of dry resin. produced water. Cations are replaced with hydrogen ions using cation-exchange resins while the anions are replaced with hydroxyls using anion-exchange resins. The hydrogen ions and hydroxyls recombine producing water molecules. Hence, no ions remain in the solution.

2.2.1 Properties of Ion Exchange Resins

The ion exchange resins are generally solid gels in spherical or granular form. It consist of:
- a three dimensional polymeric network
- ionic functional group attached to the network
- counter ions
- a solvent

A strong acid, cation-exchange resins and strong base, anion-exchange resins that are fully ionized over the entire pH range are based on the copolymerization of styrene and a cross linking agent, divinylbenzene, to produce the three dimensional cross linked structure. The degree of cross linking is governed by the ratio of divinylbenzene to styrene. Weakly acid cationic-exchangers are sometimes based on the copolymeization of acrylic acid and methacrylic acid. These two cross linked copolymers swell in the presence of organic solvents and have no ion-exchange properties

To convert the copolymers to water-swellable gels with ion exchange properties ionic functional group are added to the polymeric network by reacting the copolymers with various chemicals.

Commercial ion exchangers in the hydrogen, sodium and chloride form are available under the trade names of:

- Amberlite
- Duolite
- Dowex
- Ionac
- Purolite

2.2.2 Amberlyst 15 (Wet) Resins

In this research project, Amberlyst 15 (Wet) resins is proposed to be used as the ion exchange resins as the BPPA plant is currently using the Amberlyst 15 resins as one of the catalyst in the process of the plant. This means, the plant do not need to order and buy other kind of resins just for the water treatment purpose. Amberlyst 15 (Wet) resin is a strongly acidic, macroreticular polymeric resin based on cross linked styrene divinylbenzene copolymers.

The resins have few criteria that makes it a good ion exchange resins , as below:

- long lifetime
- good stability
- currently used by BPPA in their plant process, hence save costs because the plant currently have this ion exchange resins.
- good resistance to thermal, mechanical, and osmotic shock
- low leaching
- can be used for aqueous and non-aqueous media applications.

2.3 Activated Carbon as Adsorbent

Besides ion exchange resins, activated carbons are also widely used as adsorbent. Activated carbon is a form of carbon that has been processed to make it extremely porous. With high porosity, the activated carbon have a very large surface area available for adsorption or chemical reactions. 1g of activated carbon has a surface area of approximately 500m^2. For this project, Activated Charcoal, NORIT® SA2, Decolourizing is used.

Activated carbon can be produced by two process which are:
i.) Physical reactivation

- Carbonization: Material with carbon content is pyrolyzed at temperatures in the range 600-900 °C, in absence of air (usually in inert atmosphere with gases like argon or nitrogen)

- Activation/Oxidation: Raw material or carbonised material is exposed to oxidizing atmospheres (carbon dioxide, oxygen, or steam) at temperatures above 250 °C, usually in the temperature range of 600-1200 °C.

ii) Chemical Activation

- impregnation with chemicals such as acids like phosphoric acid or bases like potassium hydroxide, sodium hydroxide or salts like zinc chloride, followed by carbonization at temperatures in the range of 450-900 °C.

There are few types of activated carbon such as Powdered Activated Carbon (PAC), Granular Activated Carbon (GAC), Extruded Activated Carbon (EAC), Impregnated Carbon, and few types.

2.3.1 General Adsorption Properties of Activated Carbon.

The surface of the activated carbon is non-polar, result of the surface oxide groups and inorganic impurities. The activated carbon can be used to perform separation without stringent moisture removal. It also has large pore volume which enables it to adsorbs more non-polar and weakly polar organic molecules than other sorbents.

The heat of adsorption, or bond strength, is lower on activated carbon than on other sorbents. This is because non-specific Van der Waals force acts as the main force for adsorption. Stripping of adsorbed molecules are also easier and results in lower energy requirements for regeneration of the sorbents.

2.3.2 Surface Chemistry and its Effects on Adsorption

Activated carbon represented as by a model of twisted network of defective hexagonal carbon layer planes (typically 5nm wide), which are cross linked by aliphatic bridging groups. Hetero atoms are incorporated into the network and are also bound to the periphery of the planes. The hetero atoms assumes the character of the functional group, which is important for adsorption in aqueous solutions and the catalytic properties of carbon. Some typical surface groups are carboxyl, quinine, hydroxyl, carnonyl, carboxylic anhydride and lactone. The acidic surface shows cation exchange properties in aqueous solutions.

If the carbon are degassed at high temperature such as 950C, in vacuo or inert atmosphere, and subsequently oxidized at room temperature after cooling, it exhibits basic character and hence acquires and anion exchange capacity. The functional groups exhibits different acid strength:

Hydroxyls > Lactones and Lactols > Carboxyls (and their anhydrides).

2.4 Types of Adsorption

2.4.1 Batch Adsorption:

Batch adsorption is usually preferred when the quantities treated are in a small amount. Let the initial feed concentration of the effluent is c_F, and the final equilibrium concentration c. Let the initial concentration of the solute adsorbed on the solid (adsorbent) is q_F, and the final equilibrium concentration is q. Thus, the material balance on the adsorbate is:

$q_F M + c_F S = qM + c_S$,
where M is the amount of adsorbent in kg and S is the volume of feed solution in m^3.

2.4.2 Continuous Adsorption:

Continuous adsorption process is widely used in the industry. For the continuous process, the fluid to be treated will be passed down through the packed beds which contains the absorbents.

2.4.2.1 Designing Fixed-Bed Adsorption Column.

The concentration of the solute in the fluid phase and of the solid adsorbent phase, changes with time and the position of the fixed bed.

When the fluid first comes on contact with the bed of adsorbents, maximum mass transfer and adsorption occurs. The fluid passes through the bed of adsorbents and the concentration of the fluid drops and reaches zero before the fluid arrives the end of the bed , as shown in figure 2.3:

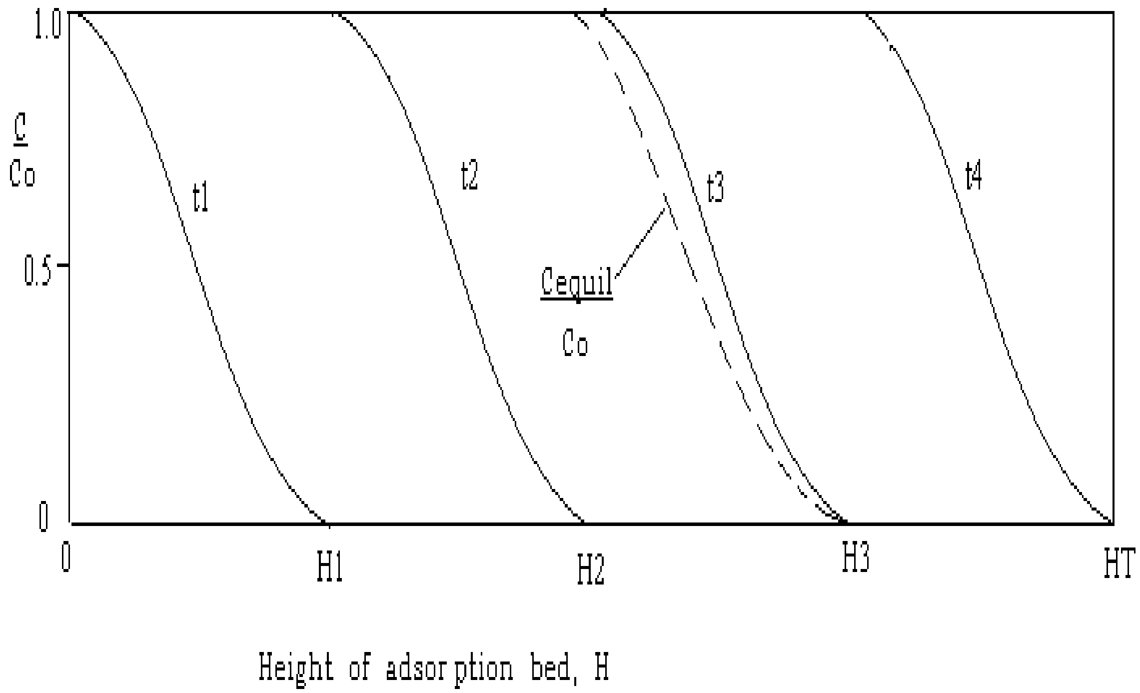


Figure 2.3: Concentration profiles at various positions and times in the bed

The concentration profile at the start t1 is shown in figure 2.3. c/c_o versus the length of the bed. c_o is the inlet concentration of the fluid (while the fluid enters the adsorption column), and c is the outlet concentration (after the fluid passes through the bed of adsorbents. After a short time of fluid passing through the bed, the solid adsorbents near the entrance of the column (top layer of the bed) will be saturated. Hence, mass transfer and adsorption takes place further from the inlet of the column which is during t2.

The dashed line t3 shows the concentration in fluid phase which is in equilibrium with the solid adsorbents. The difference of concentration between the liquid and solid phase is the driving force for the mass transfer to occur.

2.4.2.2 Breakthrough Concentration Curve

The outlet concentration of the fluid from the adsorption column remain zero until the mass transfer zone reach the outlet at t4 as shown in figure 2.4. The outlet concentration will then start to rise to c_b which is the break point as shown in figure 2.4. After the break point time is reached, the outlet concentration rises rapidly to the level c_d which is the end of the breakthrough curve. When the break point time is reached, the bed is considered ineffective.

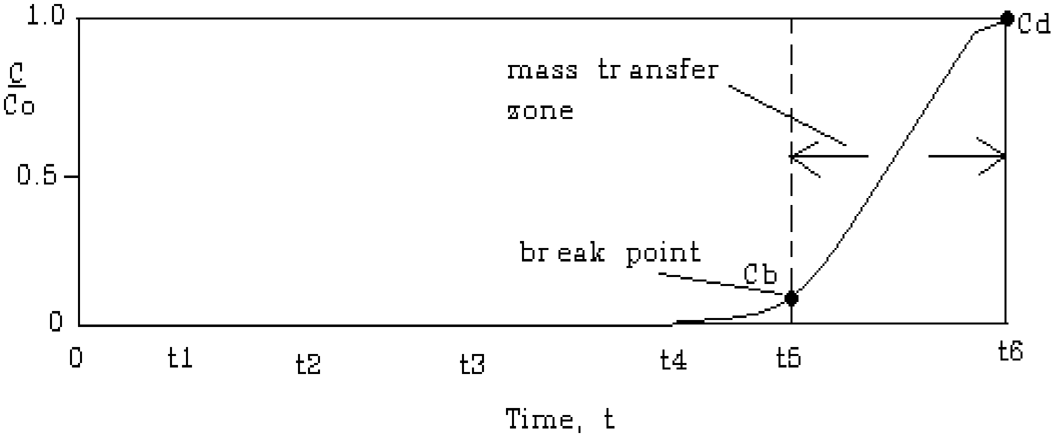


Figure 2.4: Breakthrough concentration profile in the fluid at the outlet of the bed.

The break point concentration represents the maximum amount of adsorbate that can be eliminated form the fluid. The normal value for c_b/c_o is often taken as 0.01 to 0.05. For the mass transfer zone is narrow, the breakthrough curve will be steep and most of the bed capacity is used at the break point. This makes an efficient utilization of the adsorbents and lowers the costs for regeneration.

2.4.2.3 Capacity of Column and Scale Up Design

The total stoichiometric capacity of the packed bed , if the concentration of adsorbate in the entire bed comes to equilibrium with the feed entering the adsorption column, can be shown to be proportional to the area between the curve and line at $c/c_o = 1$, as shown in figure 2.5. The shaded area represents the stoichiometric capacity of the bed:

$$t_t = \int_0^\infty (1 - (c/c_0))dt$$

t_t time equivalent to the total stoichiometric capacity.

The usable capacity of the bed up to the break point time t_b is crosshatched area

$$t_u = \int_0^{t_b} (1 - (c/c_0))dt$$

t_u time equivalent to the usable capacity or the time at which effluent concentration reaches max permissible level.

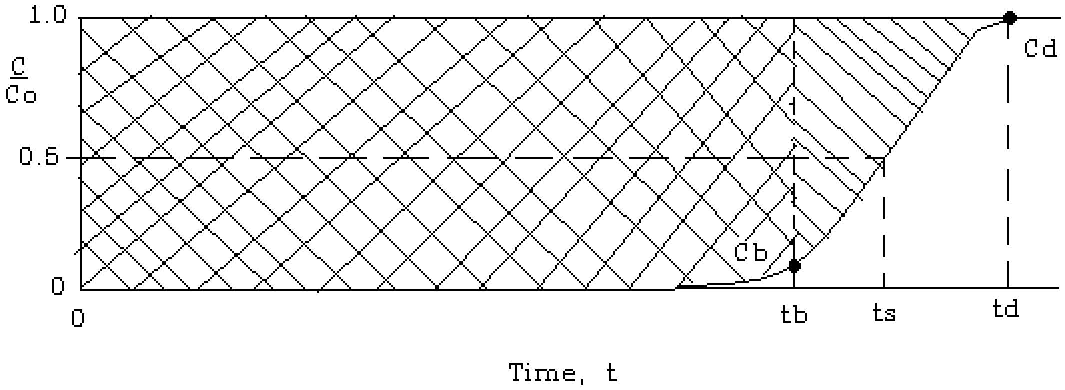


Figure 2.5: Determination of capacity of column from the breakthrough curve

The value t_u / t_t is the fraction of total bed capacity utilized up to the break point. For total bed length of H_T m, H_B is the length of bed used up to the break point.

$$H_B = (t_u / t_t) H_T$$

The length of unused bed H_{UNB} in m is unused fraction times total length:

$H_{UNB} = (1 - (t_u / t_t))\, H_T$, where H_{UNB} represents the mass transfer section and it depends on the fluid velocity. H_{UNB} is independent of the total length of the column. The H_{UNB} value can be measured at the design velocity using a small diameter laboratory column packed with the adsorbent.

The full scale adsorber bed is designed by calculating the length of the bed needed to achieve the required usable capacity, H_B, at the break point. The H_B is directly proportional to the t_b. The length H_{UNB} of the mass transfer section is added to the H_B to obtain H_T.

$$H_T = H_{UNB} + H_B$$

2.5 Models to Predict Adsorption

An unsteady-state solute material balance in the fluid is as follows for a section dz length of bed:

$$\varepsilon\,(dc/dt) + (1 - \varepsilon)\,\rho_p\,(dq/dt) = -\,\upsilon\,(dc/dz) + E\,(d^2c / dz^2)$$

ε : external void fraction of bed
υ: superficial velocity in the empty bed m/s
ρ_p : density of particle, kg/m^3
E: axial dispersion coefficient, m^2/s

The first term represents the accumulation of solute in the liquid. The second term is the accumulation of solute in the solute. The third term represents the amount of solute flowing in by convection in the section dz of the bed minus that flowing out. The last term represents the axial dispersion of solute in the bed, which leads to mixing of the solute and solvent.

2.6 Adsorption Cycle

Adsorption process can be divided in to two classes. The first one is cyclic batch system, in which the adsorption fixed bed is alternately saturated and then regenerated in cyclic manner. The second is a continuous flow system, which involves a continuous flow of adsorbent countercurrent to a flow of feed.

There are four basic methods for the cyclic batch adsorption system using fixed bed. After a bed has completed the adsorption cycle, the flow is switched to the second newly regenerated bed for adsorption. The first bed is then regenerated by any of the following methods:

i) Temperature-swing cycle: The spent adsorption bed is regenerated by heating it with embedded stream coils or with a hot purge gas stream to remove the adsorbate. The increase in temperature is used to shift the adsorption equilibrium curve and affect the regeneration of the adsorbent. The bed must be cooled finally so that it can be used in the next cycle.

ii) Pressure-swing cycle: Bed is desorbed by reducing the pressure at essentially constant temperature and then purging the bed at this low pressure with a small fraction of the product stream. Reduction in pressure shifts the adsorption equilibrium and affects the regeneration of the adsorbent.

iii) Inert-purge gas stripping cycle: The adsorbate is removed by passing a non-adsorbing or inert gas through the bed. This lowers the partial pressure or concentration around the particles and desorption occurs.

iv) Displacement-purge cycle: The pressure an d temperature are kept essentially constant as I purge-gas stripping, but a gas or liquid is used that is adsorbed more strongly than the adsorbate and displaces the adsorbate.

For activated carbon, steam stripping is often used in regeneration of solvent-recovery system. Steam stripping can be considered a combination of the temperature-swing cycle and the displacement-purge cycle.

2.7 Atomic Absorption Spectroscopy (AAS)

AAS is a technique to determine the concentration of metal elements in a sample. The electrons of the atoms in atomizer will be promoted to higher orbital for a short amount of time by adsorbing a set quantity of energy. This amount of energy is specific to a particular electron transition in a particular element. Generally each wavelength corresponds to only one element, which gives the elemental selectivity technique.

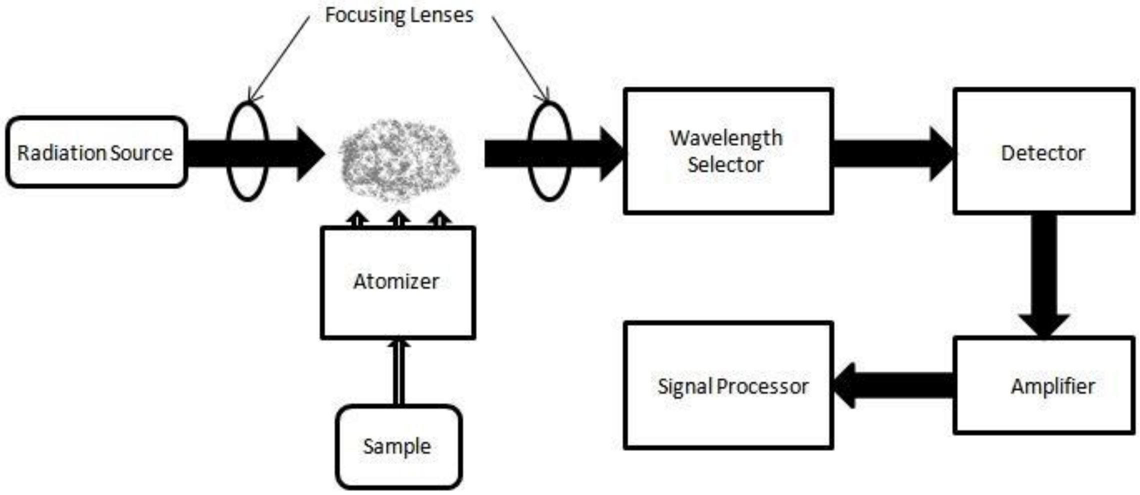


Figure 2.6: Block diagram of Atomic Absorption Spectrometer (AAS)

The elements that can be detected using the AAS machine marked in pink, is as in figure 2.7

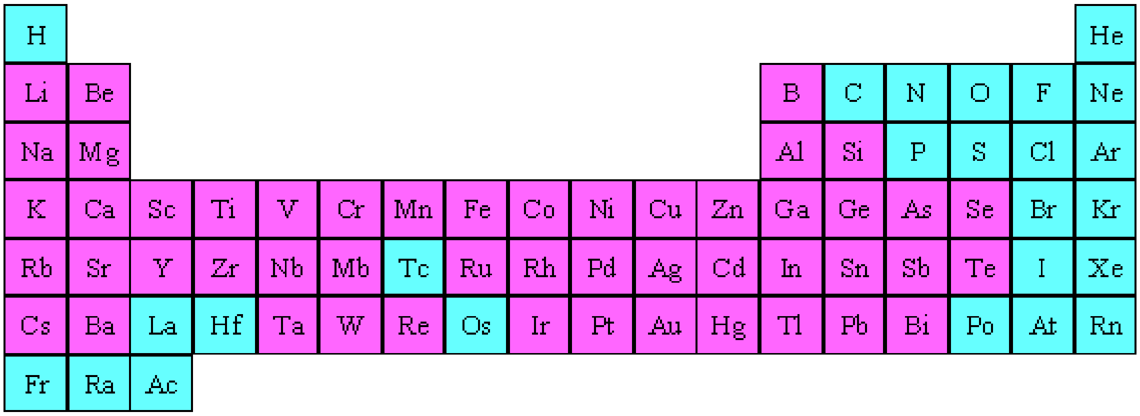


Figure 2.7: Elements that can be detected using AAS. Cr, Ni and Fe are elements that can be detected using AAS.

2.8 Industrial Applications of Sorption Process

The sorption process have few applications in the industry as summarized below:
Adsorption:

Gas Purifications:

- removal of organics from vent streams
- removal of sulphur dioxide from vent streams
- removal of sulfur compounds from gas streams
- removal of water vapour from air and other gas streams
- removal of solvents amd odors from air
- removal of NOx from Nitrogen
- removal of carbon dioxide from natural gas

Gas Bulk Separations:

- nitrogen and oxygen
- water and ethanol
- acetone and vent streams
- C_2H_4 and vent streams
- normal paraffin/ isoparaffin, aromatics
- CO, CH_4, CO_2, N_2, A, NH_3/H_2

Liquid Purifications:

- removal of water from organic solutions
- removal of sulfur compounds from organic solutions
- decolorization of solutions

Liquid Bulk Separations:

- Normal Parafin/isoparafin
- Normal paraffin/olefin
- p-xylene/otherCx aromatics
- p- or m-cymene/other cymene isomers
- p- or m-cresol/ other cresol isomers
- Fructose/dextrose, polysaccharides

Ion Exchange:

Water softening
Water demineralization
Water dealkalization
Decolorization of sugar solution
Recovery of uranium from acid leach solutions
Recovery of antibiotics from fermentation broths

2.9 Previous Research

Research have been done previously where pure Amberlyst 15 (wet) resins, and pure activated carbon is used for the ion removal (the adsorbents were not mixed). For the experiment using ion exchange resins, the most optimum ions removal needed approximately 80g of ion exchange resins. This amount of ion exchange resins is huge, and would not be cost effective for the plant.

For the experiment using activated carbon, 40g of activated carbon produced the best removal, where 100% removal of Fe(3+) and Ni(2+) ions and around 70% removal of Cr(3+) ions were achieved. This is not favorable because the Amberlyst 15 (wet) resins is not used. So the plant have to purchase the activated carbon in huge amount and the existing ion exchange resins in the plant is not utilized.

Hence the main interest of this research is to produce an adsorbent mixture to minimize the amount of activated carbon used, and to use the ion exchange resins in a moderate amount to be cost effective for the plant.

CHAPTER 3

METHODOLOGY

For this research project, the batch adsorption method is used.

3.1 Chemicals and Materials

Table 3.1: Chemicals and materials used.

No.	Chemicals	Quantity
1.	Effluents sample	1250m3
2.	Amberlyst 15(Wet) Resin	1000g
3.	Multi standard metals solution (Cr,Ni,Fe)	500m3

3.2 Equipments

Table 3.2: Equipments used.

No.	Tools/Equipment/Software
1.	Pipette
2.	Beakers
3.	Sampling bottles
4.	Weight machine
5.	Magnetic stirrer
6.	Atomic Absorption Spectroscopy(AAS)

3.3 Flow Sheet of the experiment conducted for this project

Part 3.3.1: Preparing the adsorbent mixture- The mixture as below was prepared:

mixture (a) : 0g Amberlyst + 40g Activated Carbon

Part 3.3.2: Preparing Effluents at constant volume- 200ml of the waste water sample is used

Part 3.3.3: Batch Process- Around 6ml of the wastewater sample is taken as 0 hour sample. The mixture (a) in part 3.3.1 was then mixed with the 200ml of the waste water sample. The sample was stirred at 500rpm and around 6ml of the sample were taken every 1 hour interval, for 8 hours. Total of 8 samples will be obtained at the end of the 8 hours experiment run.

Part 3.3.4: Metal Solution preparation 1ppm, 2ppm, 4ppm- Standard solutions were prepared as following: 1ppm, 2ppm and 4ppm of Chromium solutions, 1ppm,2ppm, and 4ppm of Iron solutions, and 1ppm,2ppm, and 4ppm of Nickel solutions. This standard solutions will be used to calibrate the Atomic Absorption Spectroscopy (AAS).

Part 3.3.5: Metal Testing using Atomic Absorption Spectroscopy(AAS) for Fe,Ni, Cr

The samples taken in part 3.3.3 will be analyzed for Fe^{3+}, Ni^{2+}, Cr^{3+} ion concentration in the samples, using AAS.

Part 3.3.6: Methodology repeated for the rest of the adsorbent mixtures. – The steps from part 3.3.1 to 3.3.5 will be repeated using different mixtures of adsorbents as the followings:

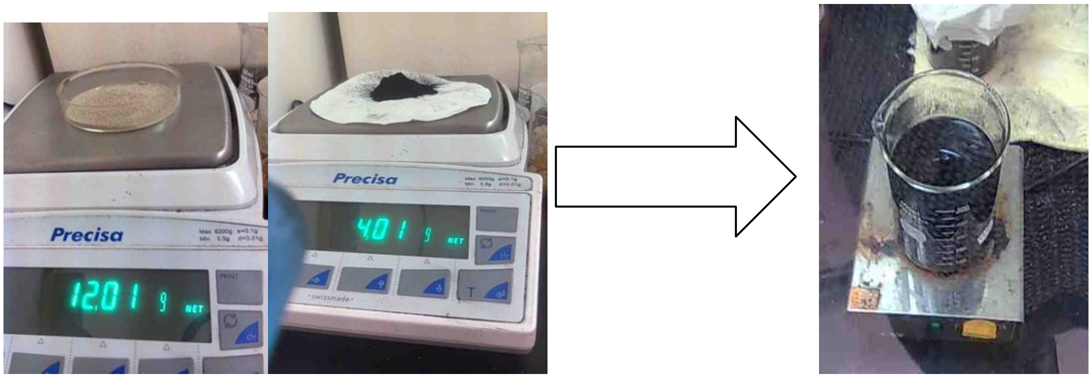
mixture (b): 10g Amberlyst + 30g Activated Carbon

mixture (c): 20g Amberlyst + 20g Activated Carbon

mixture (d): 30g Amberlyst + 10g Activated Carbon

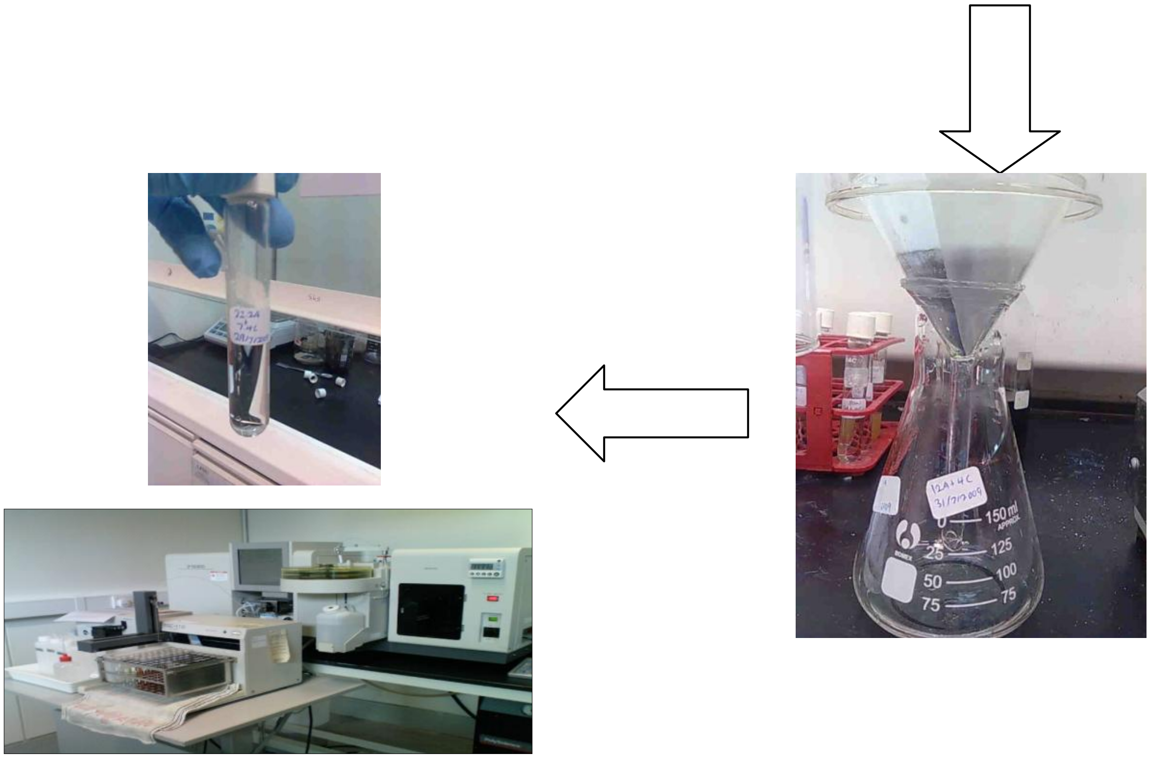
mixture (f): 40g Amberlyst + 0g Activated Carbon

Part 3.3.7: Repeatability Test: - After obtaining the best ratio of Amberlyst resins, and activated carbon, for the ion removal, this ratio of adsorbent are tested again for different amount of waste water sample, to ensure the mixture can produce same results as produced for the 200ml sample of waste water.



The ion exchange resins and activated carbon needed are weighed

The sorbents mixed with the waste water and stirred



Waste water sample sent for Atomic Absorption Spectrometer (AAS) Testing to identify the amount of metal Ions in the sample

The solution was filtered to separate the sorptions from the waste water sample

Figure 3.3: Methodology of the Experiment Done

The methodology of the experiment is simplified as the figure 3.4

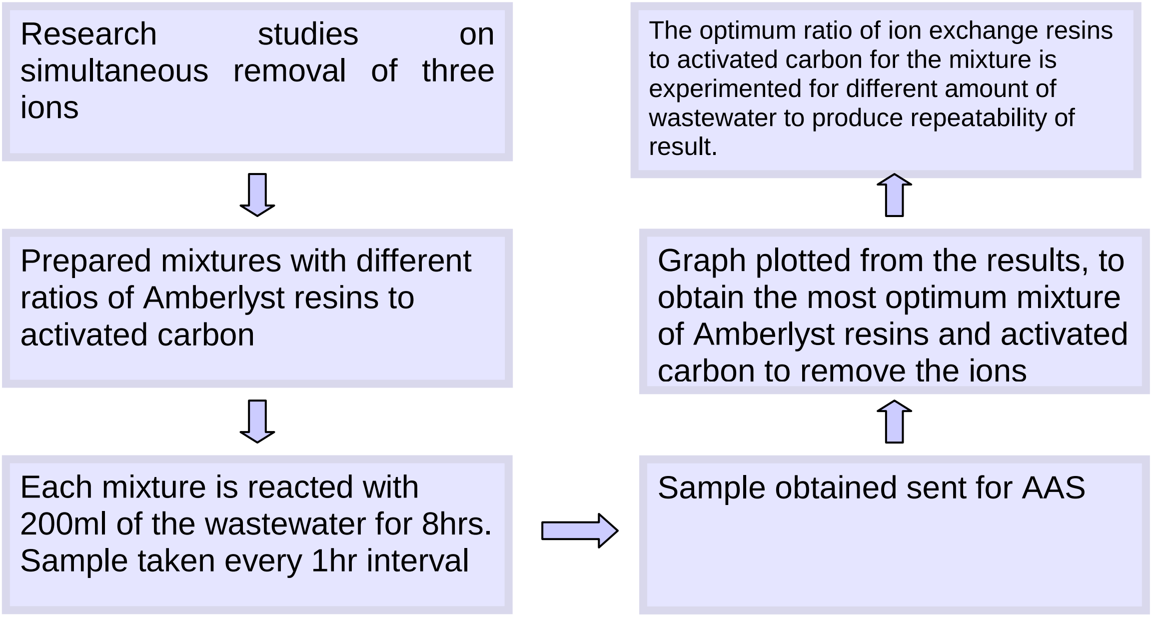


Figure 3.4: Simplified Methodology of the Project Work

3.4 Project Work Gantt Chart

					Week																	
Planned Program/Activities	**Semester Break**				**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**	**16**	**17**	**18**
Lab Work	■	■	■	■	■	■	■															
Progress Report 1								■														
Lab Work									■	■	■											
Mid Semester Break													■									
Progress report 2														■								
Pre-EDX															■							
Soft Bound Submission																	■	■				
Presentation																					■	
Hard Bound Submission																					■	

Figure 3.5: Project Gantt Chart

CHAPTER 4

RESULTS AND DISCUSSION

The amount of metal ions removed using different ratio of adsorbent mixture (results from AAS) are summarized in the graphs.

4.1 Percentage Removal of Ions Vs Time for mixtures of adsorbent

0g Amberlyst + 40g Activated Carbon:

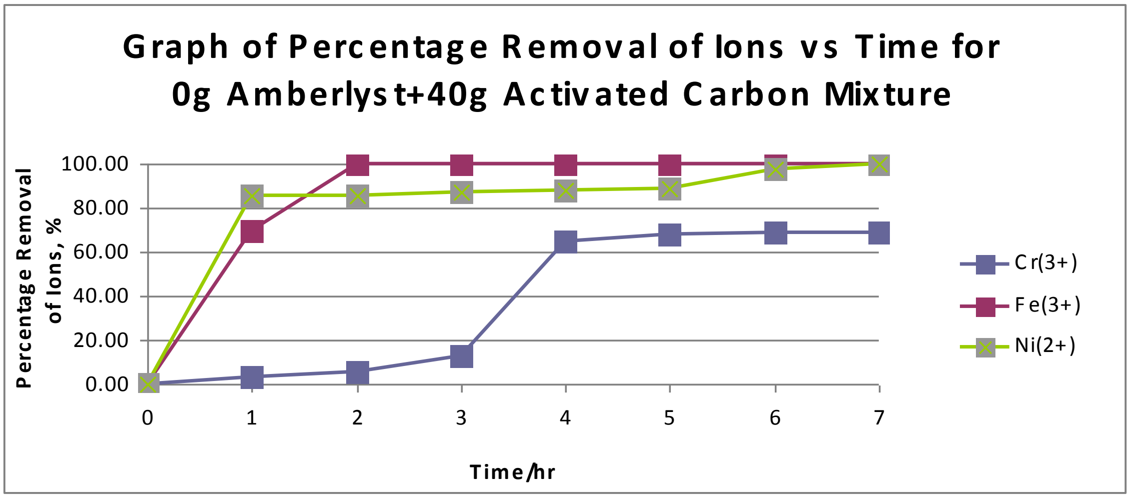


Figure 4.1: Graph of Percentage Removal of Ions Vs Time for 0g Amberlyst + 40g Activated Carbon

10g Amberlyst + 30g Activated Carbon

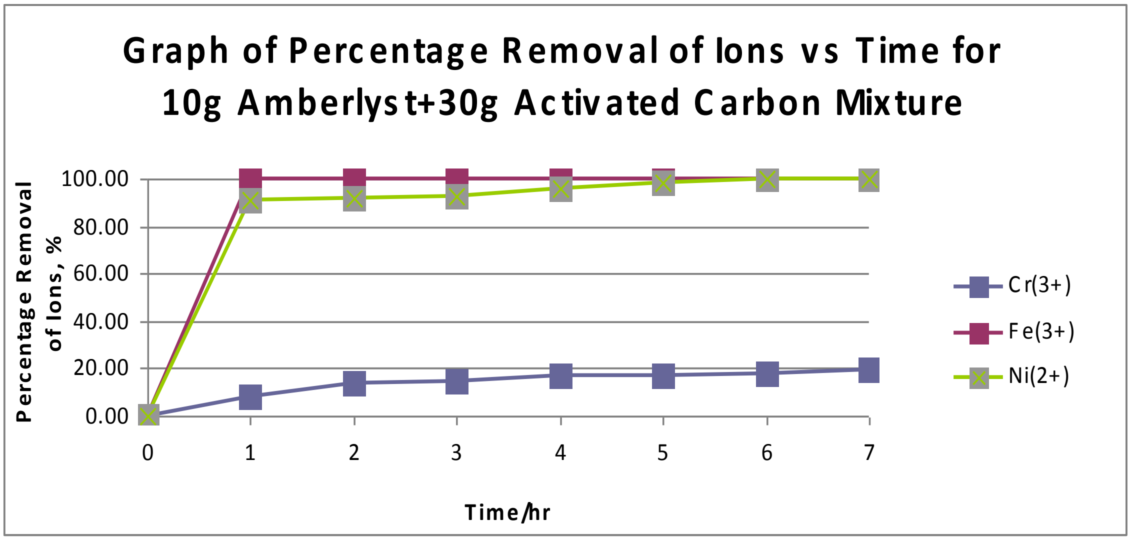


Figure 4.2: Graph of Percentage Removal of Ions Vs Time for 10g Amberlyst + 30g Activated Carbon

20g Amberlyst + 20g Activated Carbon

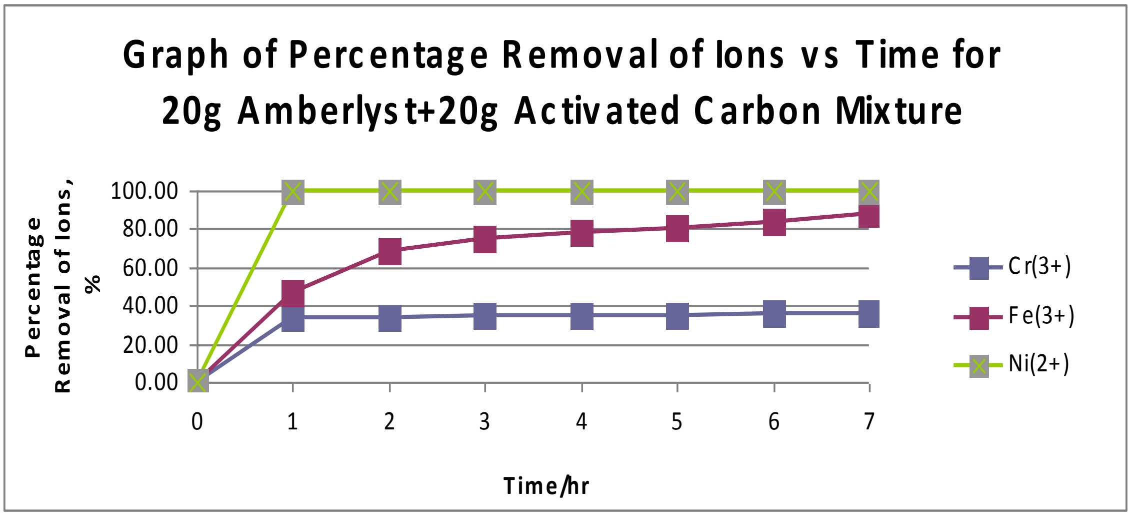


Figure 4.3: Graph of Percentage Removal of Ions Vs Time for 20g Amberlyst + 20g Activated Carbon

30g Amberlyst + 10g Activated Carbon

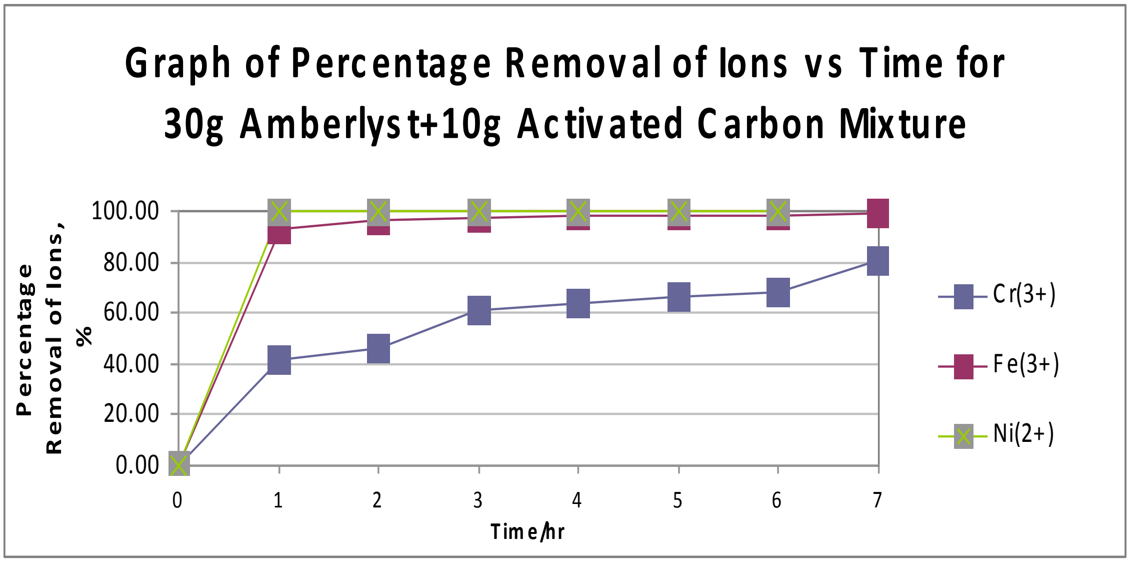


Figure 4.4: Graph of Percentage Removal of Ions Vs Time for 30g Amberlyst + 10g Activated Carbon

40g Amberlyst + 0g Activated Carbon

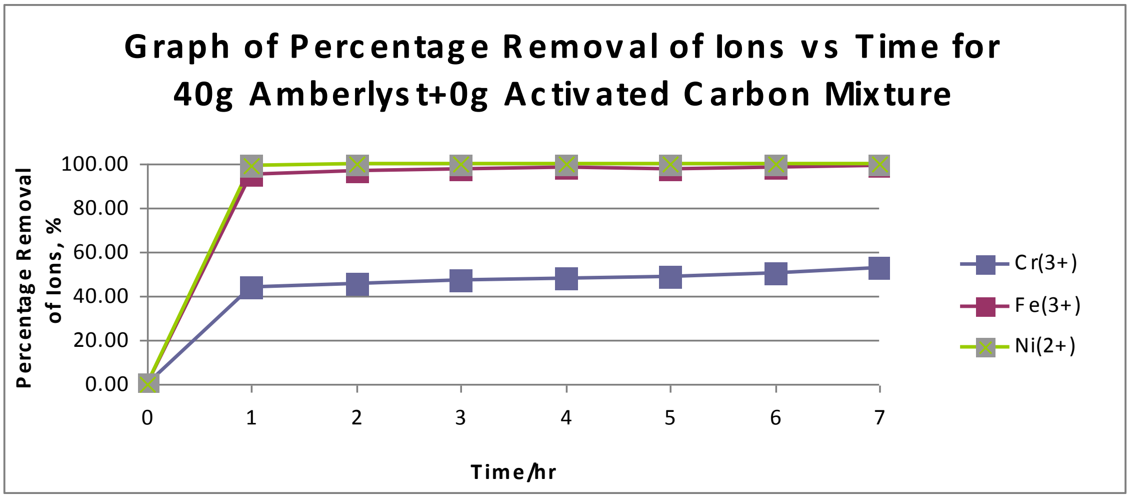


Figure 4.5: Graph of Percentage Removal of Ions Vs Time for 40g Amberlyst + 0g Activated Carbon

The results for the removal of the ions are summarized in the figure 4.6, 4.7 and 4.8:

Removal of Cr3+

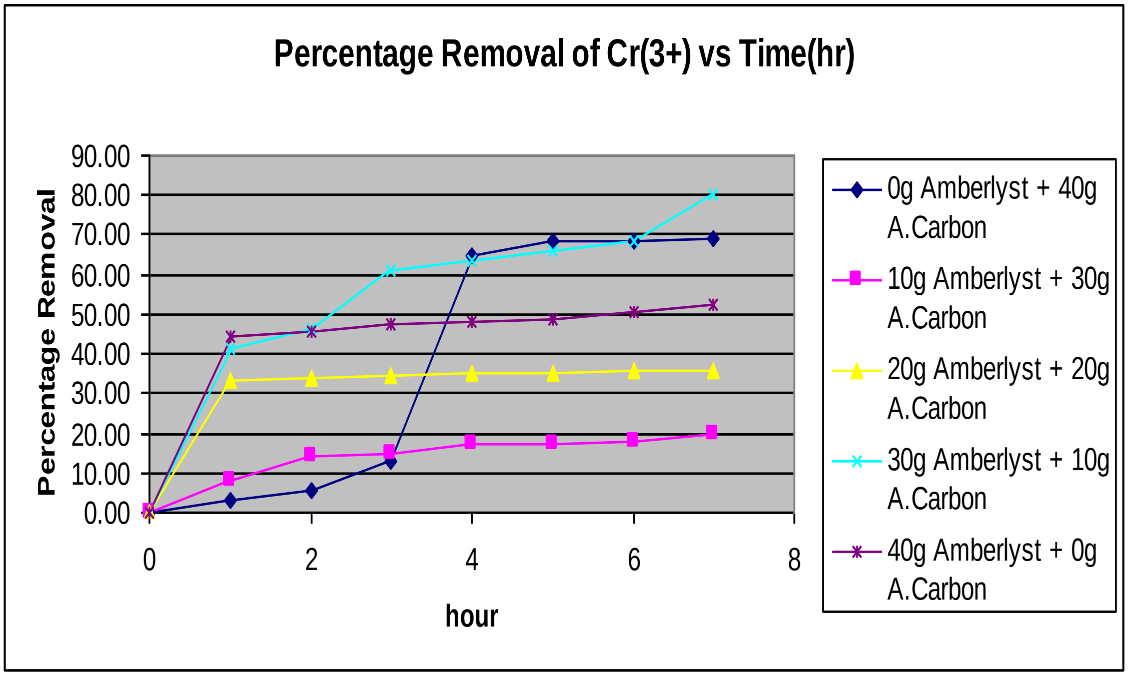


Figure 4.6: Graph of Percentage Removal of Cr (3+)Ions Vs Time (hr)

Removal of Fe3+

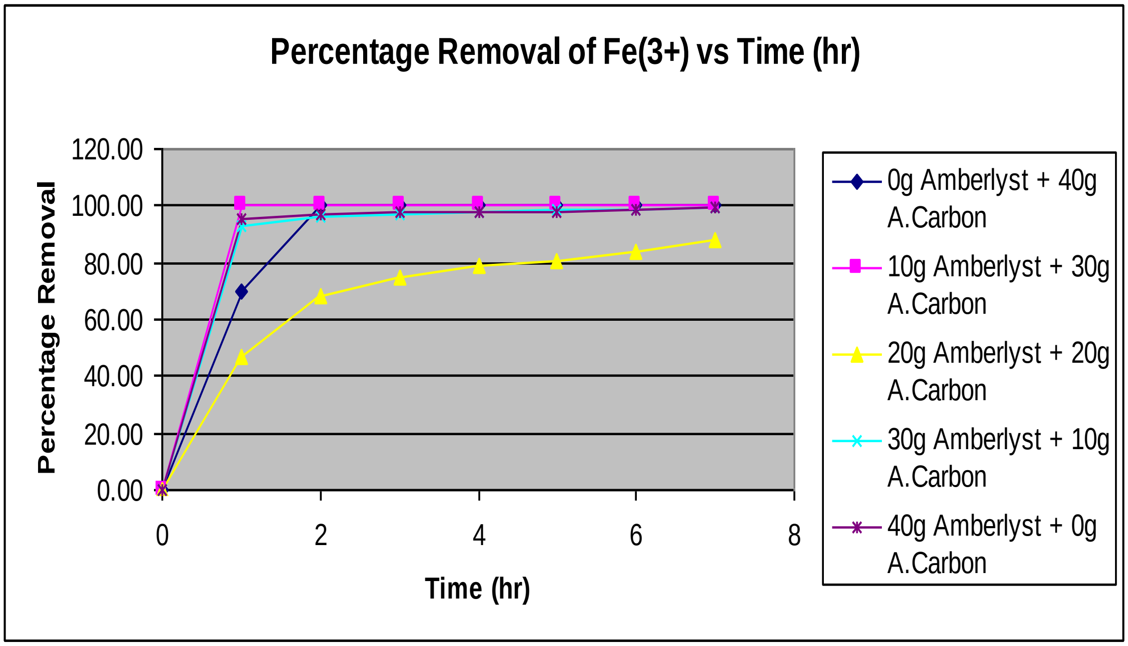


Figure 4.7: Graph of Percentage Removal of Fe (3+)Ions Vs Time (hr)

Removal of Ni2+

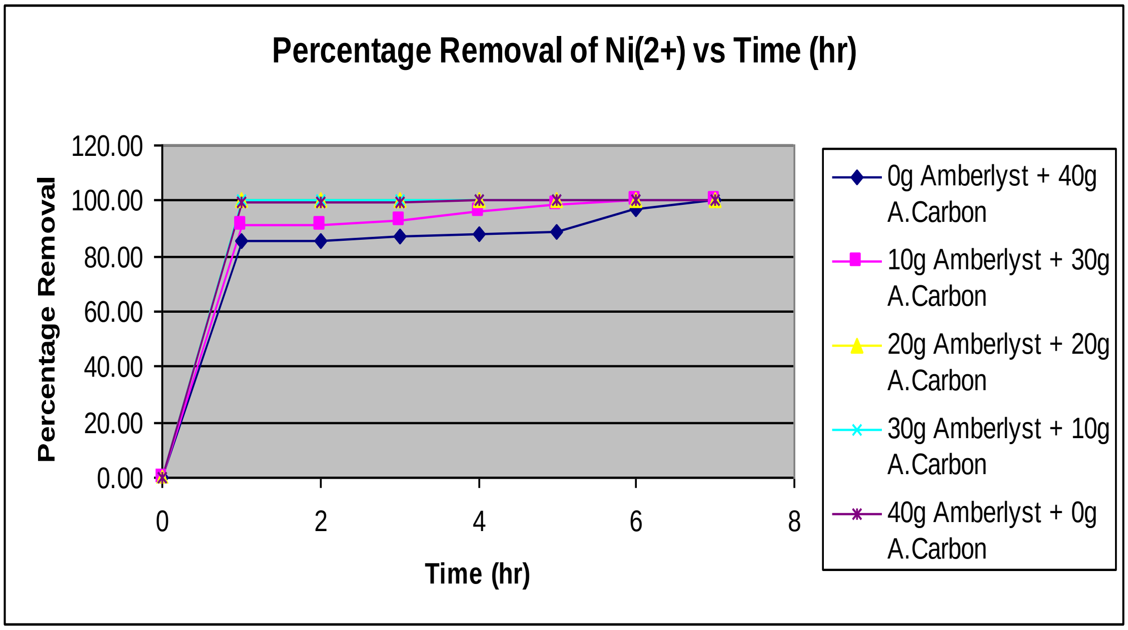


Figure 4.8: Graph of Percentage Removal of Ni (2+)Ions Vs Time (hr)

Amount of Waste Water	Mixture of Adsorbents	Ions	Percentage Removal of Ions from the Waste Water
200ml	0g Amberlyst + 40g Activated Carbon	Cr^{3+}	68.83%
		Fe^{3+}	100%
		Ni^{2+}	100%
200ml	10g Amberlyst + 30g Activated Carbon	Cr^{3+}	19.67%
		Fe^{3+}	100%
		Ni^{2+}	100%
200ml	20g Amberlyst + 20g Activated Carbon	Cr^{3+}	35.90%
		Fe^{3+}	87.80%
		Ni^{2+}	100%
200ml	30g Amberlyst + 10g Acivated Carbon	Cr^{3+}	**80.25%**
		Fe^{3+}	**100%**
		Ni^{2+}	**100%**
200ml	40g Amberlyst + 0g Activated Carbon	Cr^{3+}	52.59%
		Fe^{3+}	99.22%
		Ni^{2+}	100%

Table 4.9: Summary of removal of ions

When 0g Amberlyst + 40g Activated Carbon is used, the Fe^{3+} and Ni^{2+} can be fully removed from the waste water sample. However, the Cr^{3+} ion can only be removed around 68.83% from the waste water sample. The next mixture of 10g Amberlyst + 30g Activated Carbon removed 100% of Fe^{3+} and Ni^{2+} from the waste water sample, but could only remove 19.67% of Cr^{3+} from the waste water sample. This shows that this mixture of adsorbent is not good and have low performance in removing the metal ion from the waste water.

The 20g Amberlyst + 20g Activated Carbon removed 100% of Ni^{2+} ion, but could only remove 87.80% of Fe^{3+} and 35.90% of Cr^{3+} ion from the waste water. Next, the 30g Amberlyst + 10g Activated Carbon mixture could remove 100% of Ni^{2+}, 100% of Fe^{3+} and 80.25% of Cr^{3+} from the waste water sample. Finally, the 40g Amberlyst + 0g Activated Carbon mixture removed 52.59% of Cr^{3+}, 99.22+ of Fe^{3+} and 100% of Ni^{2+}.

From the tabulated graphs of the results, it can be seen that the Fe^{3+} and Ni^{2+} can be removed easily by almost all the mixture of adsorbents. However the Cr^{3+} ion can't be removed totally by all the mixture of adsorbents, which means the Cr^{3+} ion is the most difficult to be removed from the waste water.

From the graphs, it can be seen the mixture of 30g Amberlyst resin + 10g Activated Carbon mixture produces the highest amount of removal for Cr^{3+} ion, which is around 80% removal. Hence from all the adsorbent mixtures, the **30g Amberlyst resin + 10g Activated Carbon mixture produces the most optimal simultaneous removal of Cr^{3+}, Fe^{3+} and Ni^{2+} ions from the waste water solution.** This mixture uses less amount of activated carbon, and moderate amount of ion exchange resins, compared to the previous research as explained in section 2.9.

4.2 Repeatability Test

From the first part, it was found out that the 30g Amberlyst resin + 10g Activated Carbon mixture produced the maximum amount of metal ion removal from 200ml of the waste water. Now this mixture will be tested for a different amount of waste water to ensure that this ratio of adsorbent can produce the same results for a different amount of waste water solution. Below is the experimental steps summary and the results of the tests:

250ml of effluent + 37.5g Amberlyst + 12.5g Activated Carbon.
after 8hrs, solution filtered and 148ml solution obtained, solution tested using AAS (**T1**)

↓

148ml + 22.2g Amberlyst + 7.4g Activated Carbon
after 8hrs, solution filtered and 80ml solution obtained solution tested using AAS (**T2**).

↓

80ml + 12g Amberlyst + 4g Activated Carbon
after 8hrs, solution filtered and tested using AAS (**T3**).

RESULTS:

T1: 82% removal of Cr(3+), 100% removal of Fe(3+) and Ni(2+)
T2: 78% removal Cr(3+)
T3: 81% removal Cr(3+)
Total Cr(3+) removed: 99.25%

Figure 4.10: Summary of Repeatability Test

Hence, the adsorbent mixture are able to remove of 99.25% of Cr^{3+} ion from the waste water sample, after three rounds of treatment. This also proves that the adsorbent mixture can produce the same results when used with different amount of waste water.

From the repeatability test done, a rough idea of the type of adsorption of the ions for this experiment can be found out by plotting the graph as in figure 4.11:

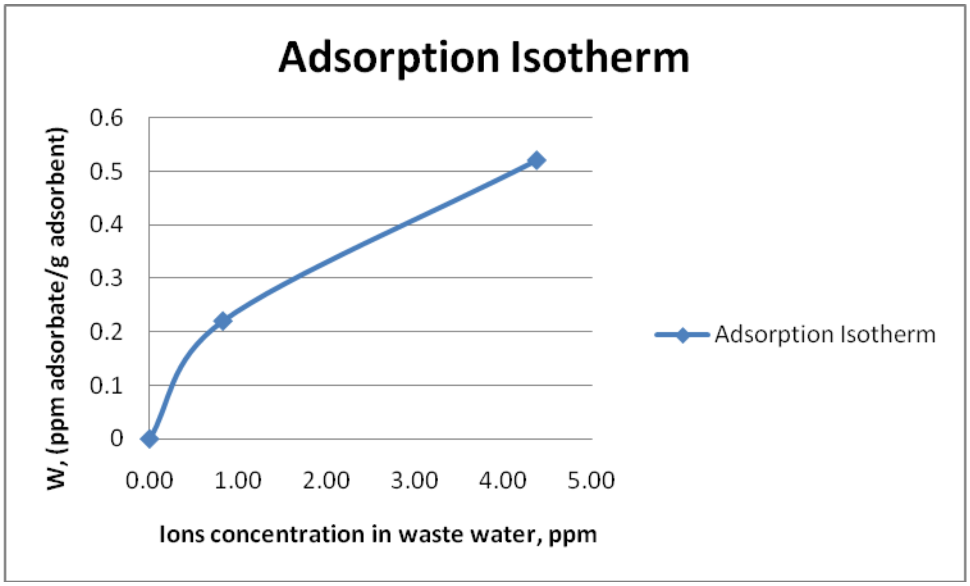


Figure 4.11: Adsorption Isotherm for the experiment done

From the graph, it can be seen a convex upwards isotherm is obtained, which is favorable. This shows that high solid loading can be obtained at a low concentration in the fluid, as explained in the chapter 2.1.3 literature review.

However, because only three sets of experiments done using different amount of waste water concentration, only a few points were obtained for the graph. More sets of experiments need to be done using various amount of fluid, to obtain more points to get a more reliable and accurate graph.

CHAPTER 5

CONCLUSION

From the experiments done, it is proved that the three metal ions Cr^{3+}, Fe^{3+} and Ni^{2+} can be removed simultaneously form the waste water, using the mixed adsorbents of Amberlyst 15 (Wet) resins and activated carbon.

The **ratio of Amberlyst resin to Activated Carbon by weight, should be 3:1**, and the **total resin and carbon mixture should be 20g for every 100ml of wastewater**, to **produce maximum simultaneous removal of Cr(3+),Fe(3+) and Ni(2+) ion** from the wastewater.

The costs of the waste water treatment can be now reduced because in house treatment can be done using the mixed adsorbent to simultaneously remove the metal ions from the waste water.

CHAPTER 6

RECOMMENDATION

There are few recommendation that can be done, to further improve and expand this study.

- more experiments should be done to get the most optimum results for removing the metal heavy metals from the waste water.
- using other types of adsorbents for the experiment for more effective removal of the metal ions from the waste water.
- analyze the impact of different variables on the experiment such as pH, temperature range of the solution and the stirring speed of the sample.
- More experiments done using different amount of waste water to obtain the adsorption isotherm
- Carrying out the experiment in continuous form to find out the amount of metal ions removed using the continuous method.
- Human error should be minimized when conducting the experiment especially when weighing the amount of adsorbent, and ensuring no contaminant contaminate the sample

CHAPTER 7

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