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Chemical Engineering Department

PRODUCTION OF DEMINERALIZED WATER FROM WASTEWATER OF PCB FACTORY BY ION EXCHANGER

A THESIS

SUBMITTED TO UNIVERSITY OF TECHNOLOGY IN PARTIAL
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By

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بسم الله الرحمن الرحيم

وَأَنْ ليسَ لِلإنْسانِ إلاّ ما سَعَى ۞
وَأَنَّ سَعْيَهُ سَوفَ يُرى ۞
ثُمَّ يُجزاهُ الجَزاءَ الأوْفى ۞

صدق الله العظيم

Dictation

To my Father and Mother with all
the love and respect
To my Wife

To my lovely Sons and Daughter

To my Family: Brothers and Sisters
And My Cousins Dr. Abbas N. and
Dr. Abdul Hussein N.
With all wishes

Mudhafar

Certificate

We Certify that this thesis was prepared under our supervision in chemical engineering department of University of Technology as a requirement for the degree of Master of Science ***in Chemical Engineering****.*

Signature:
Supervisor: ***Dr. Nahidh W. Kaseer***

Signature:
Supervisor: ***Dr. Matheel D. Al-Sabti***

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Production of Demineralized Water from Wastewater of PCB Factory by Ion Exchanger

Abstract

The aim of this study is to treat and reclamation of wastewaters from electronic printed circuit board factory (PCB) using two columns of ion exchangers in order to recycle the treated water as deionized water of high quality. Also, the study was extended to study the regeneration step of the exhausted exchangers then study the precipitation of the heavily metal content of the regenerant solutions. The reclamation of wastewater is studied in terms of two important characteristics (i.e., the adsorption capacity and breakthrough time). To study the characteristic performance of cation and anion exchangers, wastewater from electronic printed circuit board factories was subjected to a lab-scale ion exchangers of two types: the strong acid cation exchanger followed by the weak base anion exchanger. The study was divided in two major experimental investigations. The first was directed to study the performance of ion exchangers subjected to makeup aqueous solutions in terms of four different parameters (i.e., copper concentration, TDS, feed rate and bed depth). Whereas, the second experimental investigation was directed to study the ion exchanger performance towards wastewaters from PCB factory of Al-Zawraa Company in terms of three different parameters (i.e., TDS, feed rate and bed depth). Box-Wilson composite rotatable design method was adopted in designing the experiments of the two investigations (i.e., makeup aqueous solution experimental work and PCB wastewater experimental work). Adsorption capacity and breakthrough time of aqueous solution experimental investigation are measured through copper concentration between 2 to 25 ppm, TDS between 250-1250 ppm, feed rate between 0.38 to 5.34 l/h and bed depth between 5 to 70 cm. While, the measured adsorption capacity and breakthrough time of PCB wastewater experimental investigation are studied through TDS between 317 to 1193 ppm, feed rate between 0.712 to 4.980 l/h and bed depth of cation exchanger between 9.4 to 60.6 cm and anion exchanger between 14.3 to 91.0 cm.

Four 2nd order polynomial models are proposed and their coefficients are estimated using nonlinear regression analysis to correlate the two response functions (i.e., adsorption capacity and breakthrough time) with the four variables of the aqueous solution experimental investigation as well as with the three variables of PCB wastewater experimental investigation. Optimum conditions attaining maximum adsorption capacity of the two experimental investigations are determined.

In general, the experimental results have shown an efficient performance of using ionic exchanger system that consists of two columns containing a strong acid cation exchanger as (H^+) and the weak base anion exchanger as (OH^-) in order to remove the heavily toxic metals and their ions and all other cations and anions of the dissolved solids.

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Nomenclature

Symbol	*Definitions*	*Units*
B	Bed depth	cm
B. V.	Bed Volume	-
C_c	Copper concentration	ppm
C_T	Concentration of total dissolved solids	ppm
DVB	Divinyl benzene	
F	Flow rate	l/h
h	time	h
n	No. of experiment	-
PCB	Printed circuit board	-
TDS	Total Dissolved Solids	ppm
X'_1, X'_2, and X'_3	Coded variables for Al-Zawraa Wastewater experimental investigation represent TDS concentration, flow rate and bed depth respectively	-
X_1, X_2, X_3 and X_4	Coded variables for aqueous makeup experimental investigation represent Cu^{+2} concentration, TDS concentration, flow rate and bed depth respectively	-

Chapter
1

Chapter One

Introduction

Chapter One

Introduction

The most common deionization system is the strong acid cation followed by weak base or strong base anion. The product water has a pH between 7-9 and total dissolved solids concentration as low as or lower than ordinary distilled water with effluent sodium 0.5-10 ppm (Alan and Don, 2000; Galaxy Inc., 2000; Francie, 1994). Ion exchanger is a chemical treatment process used to remove unwanted ionic species from wastewater. As the name implies, ion exchange works by exchanging undesirable cations or anions in solution with less harmful ones. The ions are not destroyed but rather are removed from the waste stream and concentrated on the resin, where they can be more easily handled (Paul, 2000; Danny, 1999; Bolto and Pawlowski, 1987). The exchange reaction is reversible, and the exchange contaminates can later be removed from the resin, making the resin available for use. Ion exchange resins consist of inorganic minerals with a deficit of positive atoms within the crystalline structure, known as zeolites, or synthetic organic polymeric materials that have ionizable functional groups, such as sulfonic, phenolic, carboxylic, amine, or quaternary ammonium. In solution, salts separate into positively charged cations and negatively charged anions. Deionization can reduce the amounts of these ions to very low levels through the process of ion exchange (Kevin, 1995).

Customarily, cations are removed by cation exchange resin. It replaces sodium, calcium, copper, magnesium and other cations with hydrogen ions (H^+). This exchange produces acids, which must be removed or neutralized by anion exchange resin. Two general types of anion resin are used for deionization: weak base resin and strong base resin. Weak base resin adsorbs strong acids, while strong base resin exchanges chloride, sulfate and alkaline anions for hydroxide ions (OH^-). The hydrogen ions from the cation exchange process combine with the hydroxide ions from the anion exchange process to form water (Galaxy Onnet Inc., 2000). Because the deionization process is so effective, the water quality is usually measured by the water's resistance to electric current (in Ohm-cm).

In general, the following factors influences operating capacity, including water composition, ion exchange types and quantities, regeneration levels, flow rates, water temperature, and method of regeneration, i.e., co-flow or counter flow (Diaion, 1995; Francis, 1994; Kunin, 1963).

Printed circuit boards are electronic circuits created by mounting electronic components on a nonconductive board, and creating conductive connections between them. The conductive circuit is generally copper although aluminum, nickel, chrome and other metals are sometimes used (Higgins, 1991; Jacobs Group, 1990; Kirsch and Looby, 1991). The latest and greatest thing over the last three to four years is recycling water from printed circuit board (PCB) plants (Robert, 1998).

From practical viewpoint, this study is going to minimize the volumetric discharge containing the metals as well as the costs of purifying large quantities of wastewater from Al- Zawraa Company. A complete reclamation and wastewater treatment is designed to investigate. In order to simplify the process descriptions, the experimental work is divided into three sub-systems:

(1) Rinse water reclamation experimental work (i.e., purifying the contaminated rinse waters and wastewater from electroplated baths to a quality of approximately 10-20 μs/cm).

(2) Makeup water experimental work to replenish rinsing water that discharged to waste treatment system.

(3) Wastewater treatment experimental works where all toxic wastewaters from rinsing water reclamation are treated.

Furthermore, the study is directed to consider the reclamation system through characterizing the performance of two bed deionizers (cation and anion beds) to remove the ionic contaminants and not less than 95 % of wastewater is re-circulated to company storage tanks. In addition, the study will take in its consideration treatment of high metals content regenerant solution outcoming from reclamation system to minimize its quantity.

Chapter
2

Chapter Two

Literature Survey

Chapter Two

Literature Survey

Ion–exchange resins are insoluble polymers, which have active iongenic groups covalently bonded to the polymer. The fixed groups are either permanently ionized so that they always posses a formal charge, or are capable of ionization or acceptance of protons to form the charged side. The resin interacts with mobile ions from an external solution. Ion of opposite charge to that on the resin, and which are exchanged by the resin, are known as counter–ions, while ions of the same charge as that of the exchange sites are known as co–ions. The polymeric network of the resin is known as the matrix (Kevin, 1995; Bolto and Pawlowski, 1987). Those ion–exchange resins of exchanging cations are called cation exchangers, and resins capable of exchanging anions are called anion exchangers (Rohm and Hass Co., 1999). Certain resins can exchange both cations and anions, and are termed amphoteric ion exchangers.

The first systematic studies of ion exchange were carried out on naturally occurring inorganic ion exchangers, but they were soon overshadowed by the much greater interest shown in organic resins, since industrial applications of ion exchange now rely heavily upon the latter materials. The greater stability, higher capacity and ability to control their synthesis to yield products with reproducible properties has meant that organic ion exchangers have largely, but not entirely, displaced their inorganic counter parts in modern technology (Robert, 1980).

2.1 Basic Structure of Ion Exchange Resins

Ion exchange resins are synthetic resins having a chemical structure based on cross-linked three–dimensional polymer molecule into which functional groups such as sulfonic acid and quaternary ammonium are introduced (Scott, 1995; Diaion, 1995). Most of the polymer basis used for ion exchange resin is copolymers of styrene and divinylbenzene (DVB). The cross linked copolymer is synthesized by mixing styrene, which has only vinyl group, with DVB, which has two vinyl groups, and carrying out a suspension polymerization in water. Fig (2.1) illustrates a model synthesis (Diaion, 1995).

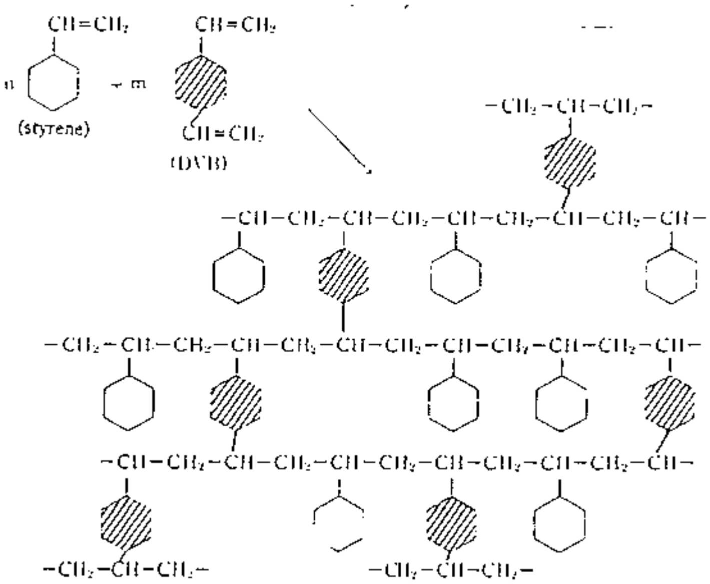


Fig (2.1) Chemical structure of styrene divinyl benzene copolymer.

The ion exchange resin is then manufactured by introducing functional groups into this copolymer matrix by means of chemical reactions. The ion exchange groups introduced, for example sulfonic acid ($-SO_3H$) or quaternary ammonium ($-N^+R_3OH^-$), are chemically bonded to the polymer, and as they cannot move freely, they are known as fixed ions (Diaion, 1995; Ullmans, 1989).

The properties of the resins are determined by the ion –exchange groups present on the matrix. In general, these may be divided into three groups: cation exchangers (strong acid or weak acid groups); anion exchangers (strong base or weak base groups); specific ion exchangers (selective chelating groups). However, the chemical activity of the exchange sites can be modified by the nature of the matrix (Bolto and Pawlowski, 1987; Robert, 1980).

The organic ion exchangers have an essentially homogeneous distribution of water throughout the resin matrix, as depicted in Fig (2.2). The resin swells under some exchange conditions and the imbibed water causes an increase in the distance between the polymeric chains (Diaion, 1995; Kirk-Othmer, 1982).

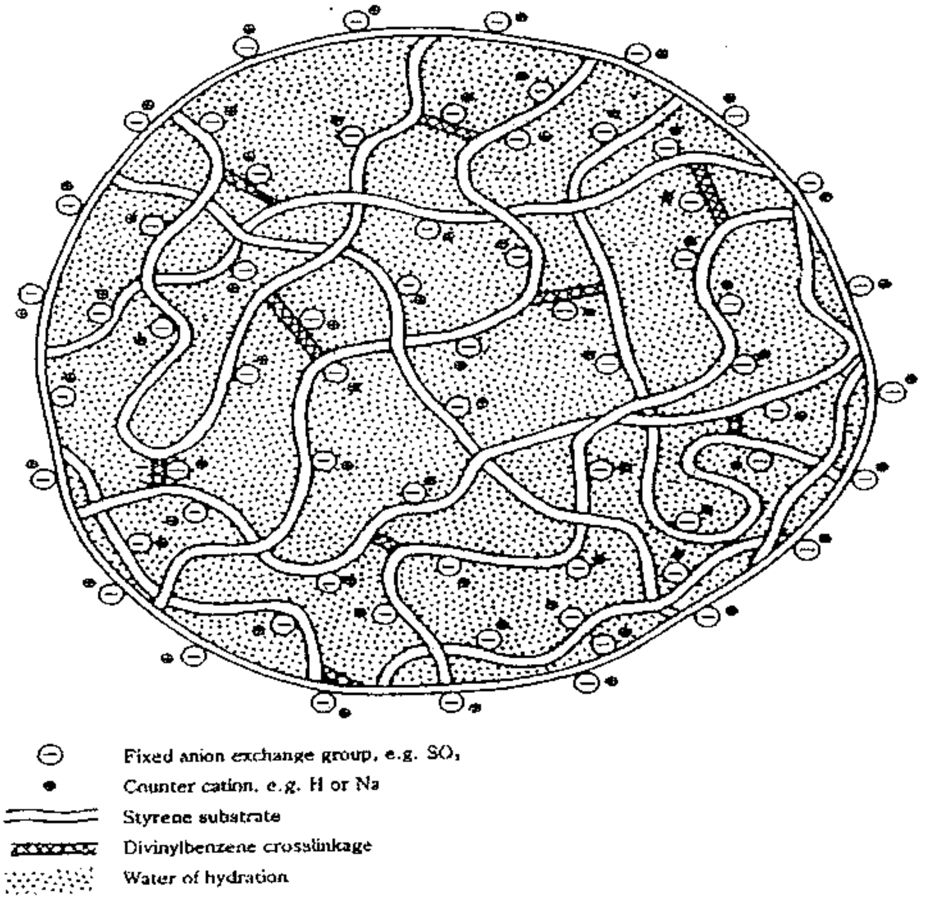


Fig (2.2) Structural model of ion exchange resin

The ability to swell depends on the degree of cross-linking, which for polystyrene resins can vary between 2 and 20 % DVB. High degree of cross-linking makes the resin less susceptible to swelling, but more brittle in nature. It is not possible to achieve an even distribution of cross-links throughout the resin matrix (Bolto and Pawlowski, 1987, Bungah, 1995). The cross-linking is less uniform when the resin bead is large, and this inhomogeneity causes some problems in practical use of the resin. With some regions within a resin bead having a different swelling ability to other regions, internal pressures result and the bead fractures. The existence of a region of denser cross linking can also hinder the release of large organic molecules form the bead during regeneration of the exhausted resin (Kevin, 1995; McKetta, 1988). In general, the progress of ions through a resin bead is retarded by the matrix polymer (Bolto and Pawlowski, 1987; Diaion, 1995). This limitation is less when there are fewer cross-links and the bead is more swollen. However, resins having a low degree of cross linking are not physically strong and more sensitive to an oxidizing environment, more highly cross linked resins are more stable in every respect, but then the tighter net work in the matrix provides a greater resistance to diffusion, and hence lower rates of ion exchange. Resins are, therefore, generally used with a cross linkage of about 8 % (Kunin, 1963; Helfferich, 1962; Diaion, 1995). As away around the dilemma resins having a macro porous structure were developed by appropriate synthetic method based on having a solvent present during the polymerization so that the growing polymer chains are precipitated in the form of micro spheres, as illustrated in Fig (2.3). The resin bead is now heterogeneous and consists of two phases: homogeneous regions containing matrix and water, and water–filled voids between these regions (Ullmans, 1989; Bolto and Pawlowski, 1987; Diaion, 1995).

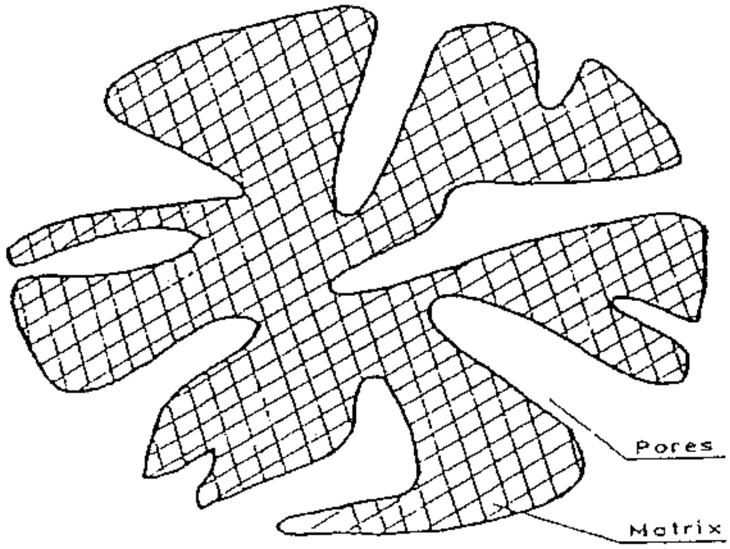


Fig (2.3) Schematic picture of macroporous ion-exchange bead

Ions entering the resin can now do so via the large pores, and the amount of matrix they must diffuse through is much less than in a gel–type resin. A more highly cross-linked matrix is employed (and is part of the synthetic requirements) which means that the bead is mechanically stronger and is less susceptible to penetration by large organic molecules, which cause fouling problems (Russell and Heyden, 1978; Kunin *et al*, 1984). As the resin bead contains a second discrete phase in the form of voids, the amount of matrix and hence the number of exchange sites is lower on a volume basis. Also, the quantity of chemicals required for regeneration of the resin is increased (Bolto and Pawlowski, 1987).

Improved rates of exchange can be obtained by using smaller particles which make a larger surface area available (Diaion, 1995). Powdered ion exchangers are employed in what has been termed precoat filters, which combine in one unit the operations of filtration and demineralization. They are successfully employed in the removal of trace amounts of impurities in condense water in the power industry (Bolto and Pawlowski, 1987). Because they are very fine particles (even though they are present in a coagulated form) they cause a high-pressure loss in operation, and must be employed a 1 cm layer on specially designed candle filters. Being a powdered mixture of cation and anion exchange resins, it is not practical to regenerate them, so they are employed on a throwaway basis. Because of the high-pressure loss, which would result, they cannot be employed in a column system (Kunin *et al.*, 1984; Purolite, 1995).

The properties of ion–exchange resins are dominated by their chemical composition and the nature of the exchange groups present on the matrix. The physical nature of the matrix can only modify these properties to a minor extent.

Weak acid resins posses carboxylic acid (-COOH) groups as the functional species (Rohm and Hass Co., 2000; Purolite, 1995). Being of weak electrolyte character, the carboxylic acid groups dissociate to varying extents depending on the pH level (Robert, 1980). They are essentially undissociated at pH 3 and below, dissociation increasing with increase in pH until by pH 10 they are fully dissociated. For example, the sites available at pH 11 for Duolite C–433 are double those available at pH 8 (Simpson *et al*, 1983). The weak acid resins in most common use today are copolymers of acrylic or methacrylic acid with DVB (Diaion, 1995; Purolite, 1995). Whereas, resin exchange anions like Cl^- or SO_4^{-2} are classified into strongly basic anion and weakly basic anion exchange resins depending on the strength of their basicity. Resins which have a quaternary ammonium group $-N^+R_3OH^-$ as the exchanging group, connected to the aromatic rings of styrene–DVB copolymers via a methylene group, dissociate in the same way as strong alkalis like NaOH or KOH and exhibit strong basicity. The quaternary ammonium exchange group of these resins is so strongly basic that it dissociates into $-N^+R_3\ OH^-$ not only in acidic but even in alkaline solutions. These are strong electrolyte groups, which remain ionized at all pH levels (Diaion, 1995). Strongly basic ion exchange resin may be classified into two types, i.e. type I with a trimethyl ammonium group $R–N^+(CH_3)_3$ and type II with dimethylethanol ammonium group $R–N^+(CH_3)_2 – CH_2CH_2OH$ (Hellfrich, 1962; Simpson *et al.* 1983). Resins with tertiary ($-NR_2$), secondary ($-NHR$) or primary ($-NH_2$) amino groups as exchange groups exhibit weak basicity, and are, therefore, known as weakly basic ion exchange resins (Bolto and Pawlowski, 1987; Diaion, 1995; Purolite, 1995; Rohm and Hass Co., 1999).

The exchange capacity of weakly basic ion exchange resins is also related to the ion concentration of the influent water. The more acidic the pH of the influent water, the better the dissociation of the exchange groups and the higher exchange capacity (Hellfrich, 1962; Kunin, 1963; Diaion, 1995).

2.2 Characteristic of the Organic Synthetic Ion – Exchangers

The characteristics of organic synthetic ion–exchange materials include ion–exchange capacity, ion exchange selectivity and regeneration ability (Grimshaw, 1975).

Many attempts Ongaro, 1950 have been made to explain the difference in the ion exchange behavior of the various anions and cations. The most plausible and satisfactory explanation involves the ionic hydration theory. Since ion exchange is controlled by electrostatic forces, presumably coulombic, the adsorbability of anion will depend on the relative charges and ionic radii of the two ions entering into the exchange. The order of the exchange affinities for the various ions is not unique to ion exchange systems and is quite common for the most ionic systems. In dilute solutions, the Hotmeister or lyotrophic series is obeyed; that is, Li < Na < K < Rb < Cs, My < Ca < Sr < Ba, Al < Sc < Y < Eu < Sm < Nd < Pr < Ce < La, F < Cl < Br < I (DiAion, 1995; Kunin, 1963).

Until a more rigorous and useful quantitative relationship can be established, and until our knowledge concerning the nature of the electrochemistry of solutions and the individual nature of ions is extended, the following set of empirical rules can be utilized quite effectively in formulating certain approximations:

1. At low concentrations (aqueous) and ordinary temperatures, the extent of exchange increases with increasing valence of the exchanging ion ($Na^+ < Ca^{++} < Al^{+3} < Th^{+4}$): (Diaion, 1995; Kunin, 1963; Deanne and Kathy Graczyk, 1997).
2. At low concentration (aqueous), ordinary temperatures, and constant valence, the extent of exchange increases with increasing atomic number of the

exchanging ion (Li < Na < K < Rb < Cs; Mg < Ca < Sr < Ba) (Diaion 1995; Kunin 1963).

3. At high concentrations, the differences in the exchange "Potentials" of the ions of different valence (Na^+ versus Ca^{+2}) diminish and, in some cases, the ion of lower valence has the higher exchange "Potential" (Kunin, 1963).

4. The relative exchange "Potentials" of various ions may be approximated from their activity coefficients, the higher the activity coefficient; the greater the exchange "Potential". (Kunin, 1963).

5. Organic ions of high molecular weight and complex metallic anionic complexes exhibit usually high exchange potential (Kunin, 1963).

6. As the degree of cross-linking or the fixed ion concentration of an ion exchange material is lowered, the exchange equilibrium constant approaches unity. (Kunin, 1963).

2.3 Water Treatment by Ion–Exchange Process

Practical water treatment generally is directed toward purifying the water to the degree that requires and seldom to complete purification or total removal of all impurities. By means of ion exchange, it is possible to remove dissolved mineral matter in apart, or in entity, or to quality characteristic of any water (Gustafan, 1947). The treatment of water consists of two processes: water softening and deionization of water (Weber, 1972).

2.3.1 Water Softening

Basically, the softening of water involve passing it through a column of cation exchanger capable of exchanging sodium ion for calcium ions, and magnesium ions in solution (Arden, 1968; Grimshaw, 1975; Purolite, 2000).

2.3.2 Deionization of Water

The deionization of water involve passing the water through a sulfonic acid exchanger as in hydrogen form in order to convert the salts to their corresponding acids and the resulting acid solution is then passed through the hydroxyl or free amine of weakly basic anion exchanger (Kunin, 1963; Galaxy Onnet Inc., 2000)

2.4 Waste Treatment by Ion – Exchangers

Wide variety materials may be removed from waste by ion – exchange materials. Removal of radioactive contaminates from wastes has been accepted practice for many years. Cation exchangers and anion exchangers are frequently employed to remove radioactive fission products from wastes of nuclear power plants. The specificity of phenolic cation exchanger has been used to decontaminate wastewater containing low levels of $^{137}C_s$ (Mark, 1964). Ion exchangers have been used for recovery of metals from plating baths restoring plating solutions which are contaminated with metals, and for removing of toxic metals from wastes either for reuse of water or prior discharging the water. Beohner and Mindler (1947) have conducted much work on removal of cyanides, thiocyanates, phenolic compounds, acids, and basis from wastes.

2.5 Ion – Exchange Kinetics

Ion exchangers have long been known to consist of three – dimensional polymeric or crystalline networks carrying fixed ionic groups and containing mobile, exchangeable counter – ions having charges of opposite sign; co-ions (mobile ions having charges of the same sign as the fixed ionic groups) are largely excluded from the interior by electrostatic forces as was first recognized by Warburg and Schulze (Jacob, 1966; Helfferich, 1962). Ordinary ion exchange is essentially a process which dose not involves an actual chemical reaction and in which neither co-ions nor fixed ionic groups play an active role. Counter ion diffusion, however, is subject to the restriction of electro-neutrality, as ions of species A move into solution, a stoichiometrically equivalent amount of species B must move into the ion exchanger to balance the electric charges of the fixed ionic groups. The counter ion fluxes thus are coupled with one another. The systems achieve this coupling by the corrective action of an automatically arising electric field (diffusion potential). Diffusion of ions, with an electric field as the mechanism of electro-neutrality conservation, is the basis of the current quantitative theories of ion – exchange kinetics (Helfferich, 1962).

In industrial practice where a solution flows through the resin, equilibrium is not necessarily reached and the results are influenced by kinetic considerations. The rate-determining step of ion exchange is the diffusion of the mobile ions toward, from and in the resin phase, rather that the chemical reaction between fixed ions of the resin and mobile counter ions. If a cation exchange resin with sulfonate ions is used as an example the cation concentration in the resin is much greater than that in solution (Ullmans, 1989).

However, any cations diffusing out of the resin into the dilute solution create a net negative charge in the solid phase and a net positive charge in solution. The resulting potential difference is called Donnan potential; it prevents anions from penetrating the resin (the phenomenon is called Donnan exclusion) (Ullmans, 1989). The bulk solution contains a large excess of available ions, with an effectively constant concentration. A static layer of solution, known as the Nernst film, surround the bead. The film is unaffected by flow around the bead; ion transport take place by diffusion only. High flow rates decrease the film thickness. The ion concentration is practically constant outside the Nernst film, and a concentration gradient occurs within. Diffusion through the film and in the solid phase occurs at different rates and two steps may be rate determining (Russel, 1978; Ullmans, 1989).

1- Diffusion of ion within the resin (particle diffusion)

2- Diffusion in the Nernst film (film diffusion)

In dilute solution (usually below 0.001 M), film diffusion is the rate-limiting step, as even in well – mixed systems the film around the resin particle is about 10 μm thick. In more concentrated solutions (generally above 0.1M), particle diffusion controls the overall rate (Helfferich, 1962). The slower step controls the over all ion exchange rate.

In general, forward and reverse exchange rate differ in a strongly acidic cation exchange resin. Forward exchange (uptake of Na^+ ions) is faster than reverse exchange (regeneration). Because the H^+ ion generally is more mobile, exchange is faster when the faster ion is in the resin initially. The rates of sodium – calcium exchange are several times slower (Purolite, 1995).

Diffusion in the resin phase is always slower than that in solution, due to the abstraction created by the resin matrix. Highly cross-linked materials have smaller diffusion coefficient (Purolite, 1995). At concentration up to 10 mg/L and flow rate up to 120 m/h used in water treatment, the diffusion rates through the resin mass are much greater that through the surrounding film. The film thus controls the rate of exchange, and the process exhibits film controlled kinetics. When sodium chloride solution pass through a column of resin, originally in H^+ form, the concentration of the ions under consideration in the emergent liquor exhibits variations (Purolite, 1995; Moison and O'Hern, 1959). If the flow rate is slow enough, equilibrium is established as the solution reaches a new layer of the resin.

Because the Nernst film thickness is an inverse function of flow rate, the film becomes thinner as the flow rate increases. Thus doubling the flow rate dose not means that the leakage curve is spread out over twice the distance. In film controlled kinetics, capacity depends very little on the rate of presentation of ions or kinetic load (i.e. the product of flow rate and concentration in the solution to be treated) The operating capacity of a strongly acidic resin depends mainly on the amount of regenerant. For a given quantity of regenerant, a corresponding capacity is available which is less than the total capacity (Purolite, 1995).

2.6 Operating Method (Method of Application)

There are three basic operating methods for ion exchangers. They consist of batch, column and moving bed operations (Deanna and Kathy, 1997). Although the last two methods are the more important (Kunin, 1963):

2.6.1 Batch Wise Operation

This mode of application is by far the simplest but also the most inefficient. The operation consists essentially of contacting the whole of an electrolyte solution to be treated with a mass of exchanger and then separating the two phases by means of filtration (Deanna and Kathy, 1997; Helfferich, 1962).

2.6.2 Columnar Operation

The fact that most ion exchange process are equilibrium reaction necessitating a continuous contacting of the exchanger with fresh electrolyte in order to drive the reaction to completion has made the column technique a most widely used method for conducting ion exchange reaction. Column operation is like several batch reactors in series (Kunin, 1963). The columnar operation is essentially an elaborate multiple batch wise technique in which the upper most portion of the column is constantly contacting fresh electrolyte whereas the lower portions contact the electrolyte not adsorbed by the upper exchanger. This procedure permits the exchanger bed to become fully exhausted at the top first and then gradually downward.

If a solution of an electrolyte is passed through a column of an exchanger, the course of the reaction may be followed by analyzing continuously the effluent solution. The concentration history (C/C_0) and the capacity of the column at breakthrough point depend on such factors a (1) regeneration level, (2) nature of equilibrium, (3) flow rate, (4) bed depth, (5) concentration, and (6) particle size (Kunin, 1963).

2.6.3 Fluidized – Bed Operation and Moving

Although the batch wise and columnar modes of ion exchange operation are essentially the only ones practiced commercially, other methods are now being taken into consideration of interest is the fluidized – bed operation, an operation found to be most successful in the catalytic cracking process (Kunin, 1963). Moving beds are the most economical beds of three. The principle is resin and solution flow through the system. The resin is contacted counter – currently with the exhausting stream and regenerated stream (Slater, 1974). The physical chemistry is similar to fixed bed and can also be through similarly as distillation column. The advantages of operation is, there is continuous product of uniform quality at less space, capital and labor (Deanna and Kathy, 1997). The chief problem that has hindered the rapid growth of fluidized – bed principle in ion exchange has been the mechanics of the operation. The practical design of devices for continuously circulating resins and properly distributing the resin particles in the liquid, without crushing or grinding them, has been a difficult task. However, even with these difficulties, several remarkable advances have been made and several large-scale pilot plant operations have been reported by Weiss (1953) and Higgins (1954). In addition to these, a plant–sized operation for water softening has also reported by Nachod and Schubert, 1956).

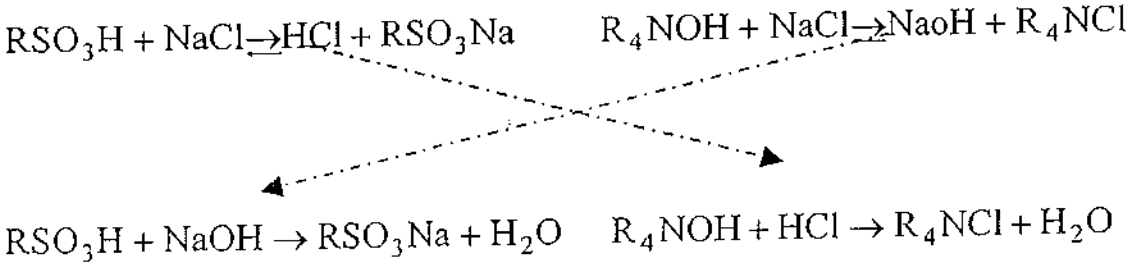
2.7 Methods of Deionizing

There are three methods for deionizing electrolyte solutions with ion exchange resins. Perhaps for lack of more suitable terminology, these methods may be designated as conventional, reverse, and mixed–bed deionization methods (Scott, 1995; Helfferich, 1962). In the first method, water or some other electrolyte solution is passed through the hydrogen form of a sulfonic acid exchanger in order to convert the salts to their corresponding acids. The acid solution is then passed through the hydroxyl or free amine form of the weakly basic anion exchanger (Rohm and Hass Co., 1999; Francis, 1994).

In the second method the various salts of the electrolyte solution are converted into their respective hydroxides by passage through the hydroxyl form of strong–base anion exchanger. The hydroxides are then passed through the hydrogen form of weakly acidic carboxyl exchanger (Kunin, 1963; Helfferich, 1962).

The last method, mixed bed, is the most recent of all. The stoichiometric mixture of the hydrogen form of a sulfonic acid cation exchange resin is merely contacted with the electrolyte solution batch wise, or the solution may be percolated through a bed of the mixed resins. Since both exchangers exhibit comparable and rapid exchange rates, deionization proceeds rapidly at a neutral pH. The reaction may be written as (Alan and Don, 2000; Francis, 1994):

$$RSO_3H + R_4NOH + NaCl \rightleftarrows RSO_3Na + R_4NCl + H_2O$$



$$RSO_3H + NaCl \rightleftarrows HCl + RSO_3Na \qquad R_4NOH + NaCl \rightleftarrows NaoH + R_4NCl$$

$$RSO_3H + NaOH \rightarrow RSO_3Na + H_2O \qquad R_4NOH + HCl \rightarrow R_4NCl + H_2O$$

In any of the three methods described, the resin acidity or basicity may be varied by choosing the desired resins according to the desired effluent water quality or resin regeneration efficiency. For example, in conventional deionization, a strong base resin could be substituted for the weak – base resin; however, substitution of a weak acid cation exchange resin. The substitutions are also possible in the cases of reverse deionization and mono bed deionization (ABB Inst., 1999).

The advantage of any of these methods over the other will depend on the application and the pH requirements during the operate at comparable efficiencies.

2.8 Conventional Deionization

The deionization of an electrolyte solution by passage through a bed of the hydrogen form a sulfonic acid exchanger and then through the bed of an anion exchanger is dependent on many variables, several of which are independent and several dependent on each other. The variables are (1) total concentration of influent, (2) ionic composition of influent, (3) regeneration level, (4) rate of flow, (5) temperature, (6) particle size of exchangers, (7) concentration of regenerant and (8) type of regenerant (ABB Inst., 1999; Thompson and McGarvey, 1948). The foregoing variables apply equally to both the anion and the cation exchangers, and a discussion of them must consider the role of exchangers separately.

2.8.1 Cation Exchanger

In discussing the role of the cation exchanger in deionization, there are two major points that are of interest, the leakage and the capacity of the unit. The former determines primarily the quality of the deionization, and the latter the cost or efficiency. Since the salts present in the water or solution must first be converted to their corresponding acids as effectively as possible, and since the conversion is in equilibrium reaction, the completeness of conversion will depend on the nature of both the anions and the cations (Francis, 1994).

The degree of acid formation, for a given anion, follows the usual exchange potential series; that is, the ability to replace hydrogen ions constitutes the series $Li^+ < Na^+ < K^+ = NH^+ < Rb^+ < Cs^+$; $Mg^{+2} < Ca^{+2} < Sr^{+2} <$; $Na^+ < Ca^{+2} < Al^{+3}$. For any given cation, the ability to replace hydroxyl ion follows the series $Cl^- <$

$SO_4^{-2} = NO_3^- < H_2PO_4^- < HPO_4^{-2} < HCO_3^- < CO_3^{-2} < OH^-$. The cations of the influent that have not been exchanged for hydrogen ions are designated as "leaking" cations, and the concentration of these cations is designated as "leakage". It is quite obvious that, for a given leakage, the capacity realized will vary according to the lyotropic series (Danny, 1999). Another factor influencing the leakage is the concentration of the influent solution. As the concentration increases, the leakage of salt increases. The rate at which this leakage increases with concentration diminishes with increasing cation valence and decreasing acidity of the acid corresponding to the anion of the salt. At a constant concentration and for a given cation, the leakage or incompleteness of conversion of various salts will be in the order $Cl > SO_4 > C_2H_3O_2 > HCO_3$ (Kunin, 1963).

A most important variable that leakage is dependent on, is the degree of regeneration. Although of some importance to all cations, the dependence of leakage on regeneration is quite marked for the monovalent cations. It is interesting to observe that during all exhaustion cycles the leakage is at the beginning of the cycle and drops to a minimum, rising again in the vicinity of the breakthrough point. This pattern holds true for all regeneration levels except those that are sufficiently high to insure complete regeneration. The mechanism for the leakage pattern at low regeneration levels is quite simple. If the column is not fully regenerated portion of the bed, the exchanged hydrogen ions migrate downwards and come in contact with the unregenerated lower portion. They then exchange for the sodium ions that appear in the effluent as leaking cations (Kunin, 1963).

Leakage may be controlled in one of several ways. First, it may be lessened by the use of larger quantities of regenerant. Second, leakage may be decreased by means of counter current operation. Third, since the leakage is a result of an unregenerated band, mixing of the bed after regeneration will dilute this band and thereby reduce the leakage during the exhaustion that follows (Remco, 2001).

Although the type and the concentration of the regenerant have not been considered in the forgoing discussion, these factors have a pronounced effect on the efficiency of the regenerant solution and therefore on the degree of regeneration. Indirectly, then, these factors influence the leakage and breakthrough capacity. Since the regeneration of the hydrogen unit depends on the replacement of the adsorbed exhausting cations with hydrogen ions, it is quite obvious that the strength of the acid and its concentration (or activity) will have a pronounced effect. For the regeneration of exhausted resin, all strong acids (HCl, H_2SO_4, etc.) have been found to have similar regeneration efficiencies at equal concentrations. Studies have shown that at dilute concentrations hydrochloric acid is much more efficient as a regenerant than sulfuric acid for magnesium as well as calcium – saturated resins. For sodium – saturated resins, the difference in regeneration efficiencies between the two acids is slight. There is one exception to this rule: the case of the regeneration of calcium–exhausted resin with sulfuric acid. Under these conditions, as the calcium ions are replaced, they precipitate in the resin bed as calcium sulfate, which later dissolves and partially exhausts the resin through an exchange between the calcium and hydrogen ions, thereby resulting in a lower regeneration efficiency. Actually, the regeneration efficiency is equal to that obtained with hydrochloric acid. However, the secondary effect of the solution of the calcium sulfate indirectly results in an apparent decrease in regeneration efficiency. In addition to a loss in efficiency, calcium sulfate precipitates may "set up" within a column to such an extent that no reasonable flow rates can be maintained (Kunin, 1963; Remco, 2001).

The effect of regenerant concentration upon regeneration efficiency is quite marked. As the concentration increases, the efficiency increases, and it reaches a maximum at about a concentration of 10 percent. Again, the case in which calcium–exhausted resin is regenerated with sulfuric acids, demands special consideration since the position of the maximum is governed by two competing factors. Although, an increase in concentration increase the regeneration efficiency, this greater efficiency increases the extent of precipitation and thereby has a limiting effect. It is because of this effect that the maximum in this special case occurs at a lower concentration. The "susceptibility" to calcium sulfate precipitation varies with the ratio of calcium ions to total cation concentration and the capacity of the exchanger (Kunin, 1963; Remco, 2001).

The difficulties encountered with $CaSO_4$ precipitation would indicated that hydrochloric acid would be preferred. However, in view of the difficulties in handling hydrochloric acid and its much higher cost, sulfuric acid is to be preferred. It is because of these factors that most of the data has been obtained with sulfuric acid.

Three techniques have been used to minimize the effect of calcium sulfate precipitation. In the first the bulk of calcium is exchanged from the resin with sodium ions using NaCl rinse prior to the sulfuric acid regeneration (Nachod and Schubert, 1965; Rawlings and De Geofroy, 1947). In the second method the sulfuric acid is used in dilute solutions (to minimize $CaSO_4$ precipitation) at first, and gradually the concentration is increased to the desired amount. The third technique involves mixing the bed prior to regeneration. If the bed contains appreciable quantities of sodium, the calcium ion concentration within the bed will be lowered and the precipitation of $CaSO_4$ lessened (Kunin, 1963).

2.8.2 Anion Exchanger

The effluent from the cation unit contains the acids formed in the cation unit and any salt that has leaked through this unit. The choice of the anion exchanger determines to a large extent whether or not all the anions (that is, H_2S, SiO_2, CO_2, H_3BO_3) will be removed. The complete removal at SiO_2, H_2S, H_3BO_3 by means of ion exchange was not possible until a strong – base anion exchanger was available. It is advantageous to divide the role of the anion exchanger in conventional deionization into two parts, the adsorption of the strong acids and the adsorption of the weak acids (Kunin, 1963). Since the regeneration of a weakly basic anion exchanger with alkali is more efficient than the regeneration of a strong– base anion exchanger, for an economical operation it is important to adsorb as much of the acidity as possible with a weakly basic anion exchanger. The strong acids (HCl, H_2SO_4, HNO_3, and H_3PO_4) will be completely adsorbed by the weak – base anion exchanger. The acids emerging from the weakly basic anion exchanger will consist primarily of SiO_2, CO_2, H_3BO_3, and H_2S (McBrain, 1947; McKee and Johnston, 1937).

The capacity and the effluent quality of the anion exchanger unit depend primarily on the same factors as those enumerated for the cation exchanger, that is, regeneration level, regenerant type, flow rate, temperature, nature of influent, particle size, etc (Nachod and Schubert, 1965).

The nature of the regenerant is of considerable importance and determines to a major extent the economy and quality of the operation. For the weakly basic anion exchanger, regenerants frequently used are NaOH, NH_3, and Na_2CO_3. Although the order of regeneration efficiency is NaOH>Na_2CO_3>NH_3, the low equivalent weight and the cost of NH_3 have made this base an attractive regenerant. Since the weakly basic anion exchangers can be regenerated completely with but a slight excess of regenerant over that theoretically required for the amount of acid to be removed, the "loading" curve loses its significance. Utilization of regenerant levels that do not regenerate completely the bottom portions of a weakly basic anion exchange unit results in effluent characteristics that are unstable because of their high acidity resulting from the hydrolysis of this portion of the bed during operation (Kunin, 1963).

The regeneration of the strong – base anion exchange resin differs considerably from that of the weakly basic resin. Since the hydroxyl from this exchanger is a strong base, it is necessary to regenerate with a strong base. The use of ammonia as a regenerant for these exchangers is not practical at all. Although complete regeneration of a strong – base exchanger is not necessary for the adsorption of strong acids, the hydrolysis of the silicate, borate, and sulfide salts of the exchanger necessitates a complete regeneration of these ions.

2.9 Mixed – Bed (Mono bed) Deionization

The synthesis of a strong – base anion exchange resin has made possible a new and revolutionary deionization technique that super passes by for in efficiency either of the foregoing techniques. On mixing an equivalent quantity of a strongly basic anion exchanger with an equivalent quantity of a strong – acid cation exchanger, it is possible to achieve, either by batch or column operation, a complete deionization of a solution maintaining a neutral pH through the entire exchange process (Kunin, 1963).

In order to achieve deionization at neutral pH, it is essential to remove both the anion and cations simultaneously (Kunin, 1955; Alan and Don, 2000).

To accomplish this, the cation exchanger (the strong – acid type) must be mixed with the anion exchanger (a strong base). It is interesting to note that rates of exchange of both a sulfonic acid exchanger and strong – base anion exchanger are approximately the same for equivalent particle sizes, thereby permitting the exchange mixture to maintain a constant pH under non – equilibrium conditions. An effluent of 10^7 ohm-cm is not difficult to achieve in a single – column unit. Because of the nature of the two equilibria involved in the mixed–bed technique, it is possible to achieve an effluent quality of distilled water ($10^6 - 10^7$ ohm–cm) for waters of any concentration and at any regeneration level (Alan and Don, 2000).

The chief advantages of a mixed – bed operation may be summarized as follows (Kunin, 1963):

1. Space requirements are decreased.
2. Rinse requirements are lowered.
3. Effluent qualities are more independent of regenerant levels.
4. Higher capacities may be realized than are encountered in two bed systems for high – quality water.
5. Water qualities are realized in a single bed that can only be realized in an alternating series of multiple beds of anion and cation resins.

Mixed bed deionization serves as an economical and convenient way to produce high purity water. In particular, mixed bed ion exchange units have found widespread applications in the power generation and electronic component industries (Abrams and Wolff, 1982; Tittle, 1981). However, technological advances in equipment design have necessitated continual reductions in the quantities of corrosion products and dissolved solids allowed in industrial water. In certain cases, current water standards limit the concentrations of dissolved solids to less than 1ppb (Tittle, 1981; Darji, 1980).

These low impurity requirements have placed stringent demands upon the performance of mixed – bed ion – exchange units.

Mixed – bed deionization requires the use of intimate mixture of cation – and anion – exchange resins. Water with the lowest impurity levels is produced in the "hydrogen – hydroxide" mode of operation (Darji, 1980). The cation resin replaces cations with hydrogen ions, while the anion resin replaces anions with hydroxide ions. The hydrogen and hydroxide ions then combine to form water. Early investigations of mixed–bed column performance, in typical deionization systems at high flow rates and low solution concentrations, concluded that the exchange kinetics were controlled by film diffusion through the liquid layer surrounding each resin bed (Cadell and Moison, 1954; Kunin and Firsch, 1960, Kunin and McGarvey, 1951). The assumptions of a completely stagnant fluid film surrounding each particle and constant ionic diffusion coefficients were generally made for ion – exchange rate investigations. Also, exchange kinetics was assumed to cover by a linear driving force due to concentration differences across the liquid film. In mixed bed units, the mixture of cation and anion resins was considered as a single salt removing resin. Other assumptions particularly applicable to mixed bed process included irreversible exchange, no water dissociation, and equal rates of cation and anion exchange.

Moreover Helfferich (1962) and Kotaoka (1976) have shown that a neutralization reaction following ion exchange has a pronounced effect on the rate of exchange. In fact, the exchange rate may be partially controlled by diffusion to a reaction front of ions, which are not involved in the exchange process.

2.10 Operations Required for Cyclic Use

In order to use ion exchanger in cyclic column operation it is necessary that a series of basic operations be employed. These are back washing, regeneration, rinsing, and exhaustion (Nachod and Schubert, 1965; Purolite, 2000).

2.10.1 Back washing

The backwash is usually considered the first step in the preparation of ion exchanger for regeneration. This up flow washing usually takes place after an ion exchanger has completed an operating or exhausting cycle. However, there may be occasions when this step will be eliminated or will follow regeneration. Several things are accomplished by back washing. Physically, the bed of material is loosened, expanded,, and reclassified. This permits more uniform distribution of fluid on subsequent down flow operations. Dirt and other foreign matter which are removed by filtration during down flow operation are rewashed out of the bed (Nachod and Schubert, 1965; Kirk-Othmer, 1982). Thirdly, ions which tend to become distributed in the bed in band will be uniformly mixed throughout the entire length of the bed. The density of the exchange materials, the particle size, and the viscosity of the solution all have an effect on the ability of the back wash to clean the particles and displace the suspended matter to waste. Under certain conditions of stringent use, the ion exchanger may be called on to filter large quantities of suspended solids of high density.

In some cases such as in softening of lime – treated water a crust of precipitate may form on the top of the bed. Under these conditions more drastic clean – up procedures may be necessary. The use of surface or subsurface washers or air helps to break up such formations so that they may be removed more easily on back washing. In addition, the feeding of certain chemicals to control floc formation may also be helpful. For example, the use of certain polyphosphates on iron – bearing results in a less dense precipitate of hydroxide being formed which can be removed more easily by the back washing procedure (Nachod and Schubert, 1965; Rohm and Hass Co., 1999).

2.10.2 Regeneration

Following back washing the next operation is regeneration. Many regenerants may be employed depending upon the type of resin and ions to be exchanged. Sodium chloride, sulfuric acid and hydrochloric acid are the most common regenerants for cation exchangers. Anion exchange resins are generally regenerated with sodium hydroxide or sodium carbonate solutions. There are many factors with affect the degree of regeneration. These are (1) type of resin and cross – linkage, (2) type of regenerant, (3) composition of the exhausted bed, (4) flow rate, (5) temperature, (6) purity of regenerant, (7) concentration of regenerant, (8) contact time, and (9) the amount of regenerant applied (Nachod and Schbert, 1965).

In general, regenerant concentration has little effect the elution of monovalent ions if the regenerant is monovalent. The exception to this is if the volume of regenerant applied is such that adequate contact is not maintained. Elution of Divalent ions by monovalent enchanced by increasing the concentration of regenerant.

Regenerant flow rate is quite important for good regeneration. Adequate contact time must be allowed in order to obtain maximum regenerant utilization. This time will vary with the resin type and ions being removed. For example, a sulfonated polystyrene cation exchanger of 8 % cross – linkage can be adequately brine regenerated with a 30 minute contact time. The same product at 12 % cross – linkage requires 60 minutes. Chlorides, sulfates, and bicarbonates are easily and rapidly eluted from a strong base exchanger, silica removal is somewhat more difficult and approximately 90 minutes of contact time are required for adequate silica regeneration as contrasted with 30 minutes for the other ions. A special case illustrating the importance of flow rate is the regeneration of a cation exchanger with sulfuric acid. If the flow is too slow or the regenerant concentration too high calcium sulfate will form in the bed and will clog the resin. Reduction of regenerant concentration or an increase in flow rate will eliminate this problem (Nachod and Schubert, 1965; ABB Inst., 1999).

The extent to which the elution is carried out will determine the efficiency of the regeneration and also the operating capacity. If sufficient regenerant is applied, all exchangeable ions will be eluted from the resin and the exchanger will be restarted to full operating capacity. This would be a very costly practice and is seldom used in industrial applications. The regeneration is usually carried out to the extent that only a portion of available capacity of the exchanger is used at a level of efficiency in keeping with the economy required of the operation. It is possible to improve on regenerant efficiency by practicing reuse or recycle of regenerants. This is particularly applicable with HCl regeneration of cation exchangers since no precipitates are formed. For recycle it is convenient to consider the regenerant as consisting of two parts. The first, which has already been used once, is passed through the exchanger first. The effluent is discarded. The second volume is a fresh regenerant, which is collected after being passed through the exchanger and recycled as the first regenerant portion for the next cycle. This technique results in considerably better regenerant efficiencies. The leakage of ions during the operating cycle is dependent upon the effectiveness or extent of regeneration after exhaustion. The weakliest ion held by the resin will be at the bottom of the bed and will constitute the "leakage". In normal water treatment practice sodium ion leaks from the cation exchanger, when operating on the hydrogen cycle, and silica from the strong base anion exchanger. The more complete the elution of these ions during regeneration the lower the leakage will be during the operation cycle (Remco, 2001; Purolite, 2000; Nachod and Schubert, 1965).

2.10.3 Rinsing

Following the application of regenerant the exchanger bed must be rinsed from free excess regenerant. The initial period of rinsing is really an extension of the regeneration phase since the exchanger bed is still in contact with regenerant. For this reason, the flow rate normally does not exceed the regenerating flow rate. The displacement of the regenerant should not be interrupted or impeded during regeneration and rinse, particularly when sulfuric acid is used, since calcium sulfate precipitation may occur. After the regenerant has been displaced the rinse flow rate may be increased to reduce rinse time. During the final rinse a gradual improvement of effluent quality is found until all of the excess regenerant is diminated from the bed. Final rinse flow rates usually coincide with the operating flow rate. In instances where extreme quality requirements may necessitate the use of extensive rinse volumes it is possible to recycle the rinse water back to the influent and improve the operation considerably. A reduction in the amount of rinse water used is obtained and may be important in large deionizers. Outage time cannot be reduced materially by practice however, but an increase in the amount of usable effluent from the unit will result (Nacchod and Schubert, 1965; Grimshaw, 1975).

2.10.4 Exhaustion

After rinsing of the exchanger bed is completed, the operating cycle may start. This is also termed "exhaustion" since the capacity available following regeneration is used up or exhausted during this phase. When treating water, the exhaustant is commonly used to rinse the exchanger bed following regeneration. At a predetermined time the effluent is diverted from waste to use and the operating run begins. When treating a solution such as a process liquid it is often desirable to minimize dilution. This can be accomplished by draining the rinse water down to bed level before introducing the process liquid. The liquid is diluted only by the water, which occupies the void space in the bed and, when the concentration of liquid in the elute is sufficiently high, the solution is diverted from waste to recovery. This process is termed “sweetening on”. At the conclusion of the exhaustion cycle it is necessary to displace the process liquid from the exchanger bed and this is “sweetening off” (Nachod and Schubert, 1965). Dilution of the process liquid can be caused by the water occupying the void space in the bed and equipment and also added dilution can result from dehydration of the resin by the process liquid, if concentrated. When such dehydration occurs, the resin bed shrinks considerably. It is usually desirable to rehydrate the bed while in suspension so that the resin particles can be free to expand without imposing undue pressure on each other. Expansion and contraction of the resin can also occur during regeneration. The rate of expansion should be minimized to reduce particle breakage.

The flow rate at which the exchanger column is operated depends upon the viscosity of the solution being treated, the rate of exchange taking place and freedom from suspended contaminants. With high capacity cation exchangers and strongly basic anion exchangers the rate of exchange is very rapid, being completed within 3 to 4 inches of bed depth at an operating flow rate of 2.0 gpm/ft^3 (Nachod and Schubert, 1965; Rohm and Hass Co., 1997).

During the exhaustion cycle, channeling of the bed is sometimes experienced. This can be caused by suspended matter present in the solution being treated. The exchanger bed will filter out the suspended matter and, when sufficient cake is accumulated on and in the top of the bed, will crack and the solution will channel down through the path of least resistance. It is not uncommon to have the bed depress in the center during the exhaustion cycle. When this occurs the bed will pull away from the wall of the vessel holding the resin and the water will channel down the wall of the tank. Such short circuiting causes rapid exhaustion of that portion of the exchanger bed and per mature breakthrough of the solution being treated will result (Grimshaw, 1975).

The design of the equipment housing the exchanger bed exerts a tremendous influence on the success of the ion exchange process. Adequate distribution of the influent solution is necessary to preclude the possibility of disturbing the top of the bed during operation. Distribution on the effluent side is even more important since it is necessary that the solution pass through the entire bed to obtain full capacity. The distribution of regenerant over the entire surface of the bed is important to insure maximum utilization of resin and regenerant (Nachod and Schubert, 1965).

Chapter
3

Chapter Three

Experimental Work & Experimental Design

Chapter Three
Experimental work

3.1 The Preparatory Experiments

Before going through the central composite rotatable designed method, many preparatory experiments were made to determine variables and operating conditions within the reasonable ranges. These were categorized as follows:

3.1.1 Lowest Impurity and Highest Quantity of deionized water production experiments

To justify the right ratio of cation to anion ratio different experiments were made using conventional method of strong acid cation exchange column followed by weak base anion exchange column. Five different ratios of cation to anion were investigated. The lowest impurity level and the highest product of deionized water were found at ratio of cation to anion equal to 1:1.5. More information is available below and in Table (3.1).

The experimental conditions were made as follows:
Cation bed volume = 108 ml.
Cation bed height = 21.25 cm.
Anion bed volume = (cation bed volume / Ratio of cation to anion) ml
Anion bed height = (cation bed height / Ratio of cation to anion) ml
Concentration of NaCl in feed water solution is equal to 1000 ppm
Flow rate of feed water solution = 15 × Bed volume of cation

Table (3.1) Effect of cation to anion ratio on the production of deionized water

Exp. No.	Cation Volume (ml)	Anion Volume (ml)	Ratio Cation/Anion	Breakthrough Time (hr)	Quantity of Deionized Water Produced (Liter)
1	108	108	1/1	3.150	5.130
2	108	135	1/1.25	4.080	6.610
3	108	151	1/1.4	4.900	7.938
4	108	162	1/1.5	5.417	8.776
5	108	173	1/1.6	5.400	8.750
6	108	189	1/1.75	5.330	8.635

3.1.2 Regeneration Study

Seven experiments were made to determine the reasonable volumes for regenerating the cation exchanger using 4 % HCl and also for regenerating the anion exchanger using 4 % NaOH. Glass columns of diameter 2.54 cm for cation and anion exchanger were used. It was shown that the lowest impurity and the highest productivity of deionized water were obtained when regenerating the cation with 5 bed volumes of 4 % HCl solution and regenerating the anion with 3 bed volumes of 4 % NaOH solution. More information is available below and in Table (3.2).

The experimental conditions were made as follows:
Volume of the cation = 272.5 ml
Bed height of cation = 53.75 cm
Volume of the anion = 408.75 ml
Bed height of anion = 80.625 cm
Flow rate of feed water = 10 Bed volume/hr = 2.72 liter/hr
Concentration of NaCl solution = 1000 ppm

Table (3.2) Effect of bed volumes used to regenerate cation by HCl 4 % solution and anion by NaOH 4 % solution on production of deionized water

Exp. No.	(Regenerant) Bed Volume of HCl (4%)	(Regenerant) Bed Volume of NaOH (4%)	BreakThrough Time hr	Quantity of Deionized Water Produced (Liter)
1	6	4	10:5	28.60
2	5	3	10.4	28.30
3	4	3	7.5	20.44
4	3	3	6.5	17.71
5	2	2	/	/
6	1	1	/	/

3.1.3 Removal of Cations from Cation Exchanger

In this step cations including toxic heavy metal ions, e.g. Cu^{++} ions, which is the main constituent in the wastewater of the Electronic Printed Circuit

Factory of Al-Zawraa Company are removed from the cation exchanger using 4 % HCl solution with different volumes. More information is available below and in Table (3.3).

The experimental conditions were as follows:
Cation bed volume = 272.5 ml
Cation bed height = 53.75 cm
Anion bed volume = 408 ml
Anion bed height = 80.625 cm
Wastewater flow rate = 1.62 liter/hr/272.5 ml = 6 B. V./hr
Total dissolve solids (T. D. S.) = 1000 ppm
The cations concentration in wastewater are found as follow:
Cu^{+2} = 8.7 ppm, pb^{+2} = 0.33 ppm, Sn^{+2} = 0.071 ppm, Na^{+1} = 145 ppm, Fe^{+2} and Fe^{+3} = 0.003, pd^{+2} = 0

The breakthrough time was found equal to 15.3 hour at electric conductivity of 100 μs/cm (equal to 50 ppm of TDS) as prescribed in Water Analysis HandBook (HANNA, 2000).

Table (3.3) Effect of bed volumes used to removal of cations from cation exchanger by 4 % HCl Solution

Exp. No.	Bed Volume of HCl (ml/ml of wet resin)	Volume of HCl (liter)	Cu^{+2} effluent Conc (ppm)	Recovered Cu^{+2} (mg)	Na^{+1} effluent Conc. (ppm)	Recovered Na (mg)	pb^{+2} effluent Conc (ppm)	recovered pb^{+2} (mg)	Sn^{+2} effluent Conc (ppm)	Recovered Sn^{+2} (mg)	Ca^{+2} effluent Conc (ppm)	recovered Ca^{+2} (mg)
1	1	0.2725	001.430	000.3897	3600.00	981.00	11.610	3.1610	0.4000	0.1090	036.22	9.86995
2	2	0.5450	255.715	139.3650	6630.00	3613.35	11.610	6.3275	0.4000	0.2180	608.11	331.42
3	3	0.8175	232.477	190.0500	4469.00	3653.40	08.687	7.1016	1.2333	1.0082	1215.4	993.59
4	4	1.0900	190.945	208.1300	3356.05	3658.09	07.225	7.87525	1.6500	1.7985	1454.0	1584.86
5	5	1.3625	158.474	215.9200	2687.75	3662.06	05.902	8.0415	1.3200	1.7985	1619.2	2206.22

3.1.4 Precipitation Study of Toxic Heavy Metals

The five bed volumes that resulted from studying the removal of cations from cation exchanger are thoroughly mixed and the precipitation of the toxic heavy metals was studied especially for Cu^{+2} ions to concur environmental regulations before disposal to sewage. Precipitation of toxic ions was carried out using gradual addition of lime solution (CaO concentration of 20 %) and continuous mixing via mechanical stirrer at 40 rpm. In addition, the precipitation study was carried out at different (pH) values. Afterward, the final solution was left for 4 hour and sample was withdrawn to measure the concentrations of heavy metals in heavier and lighter phase. Results are shown in Table (3.4).

Cu^{+2} concentration in the first five bed volumes = 158.5 ppm
Pb^{+2} concentration in the first five bed volumes = 5.9 ppm
Sn^{+2} concentration in the first five bed volumes = 1.798 ppm

Table (3.4) Effect of pH on precipitation of Cu^{+2}

Exp. No.	**pH value**	**Cu^{+2} Concentration (ppm)**
1	2	42.34
2	4	28.09
3	8	0.25
4	9	0.12
5	10	0.2
6	11	0.47
7	12	4.95
8	13	35.3

For each sample, the analysis was repeated for more accuracy.

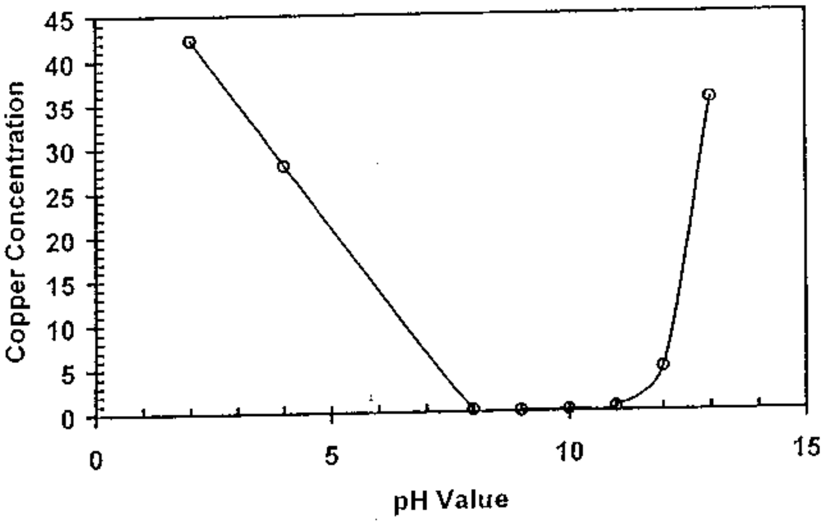


Figure (3.1) Effect of pH on precipitation of (Cu^{+2})

3.1.5 Mixed – Bed Experiments

Four experiments were carried out, two of which for conventional deionized water resulted from strong acid cation followed by weak base anion and the other two experiments with same ratios of cation to anion were carried out in mixed column. The concentrations of the solutions are 1000 and 500 ppm of TDS (NaCl solution). Fifteen bed volumes for wastewater were treated in both columns. More informations are shown in Table (3.5), in which the breakthrough point was occurred in, mixed – bed experiments before conventional experiments.

Table (3.5) Comparison study between conventional exchanger and mixed bed exchanger

Exp. No.	Vol. of Cation (ml)	Vol. of Anion (ml)	Breakthrough Time Mixed-Bed (hr)	Breakthrough Time For Conv. (hr)	Conc. of Feed Water	TDS in Effluent (ppm) Mixed-Bed	TDS in Effluent (ppm) Conventional Bed
1	107.7	161	9.25	10.75	500	1.8	8
2	107.7	161	4.58	5.47	1000	2	15

3.1.6 Study of Resulted Deionized Water with Different Bed Height Utility

Five experiments in this respect were made. The results are shown in Table (3.6). The ultimate utility percentage is resulted when the cation bed volume height reaches approximate 50 cm, and the anion bed volume height reaches approximate 75 cm.

15-bed volume of cation feed rate
Diameter of columns = 2.54 cm
TDS content of feed water = 500 ppm

Table (3.6) Experimental utilized percentage

Exp. No.	Cation Bed Height (cm)	Anion Bed Height (cm)	Breakthrough Time (hr)	Vol. of Deionized Water (liter)	Adsorption weight mg/ml wet resin	Utilized %
1	05.00	007.500	09.167	03.482	21.754	060.40
2	21.25	031.875	10.750	17.415	31.670	089.89
3	37.50	056.250	11.450	32.650	34-35	095.54
4	53.75	080.625	12.000	49.050	36.000	100.00
5	70.00	105.000	12.000	63.870	36.000	100.00

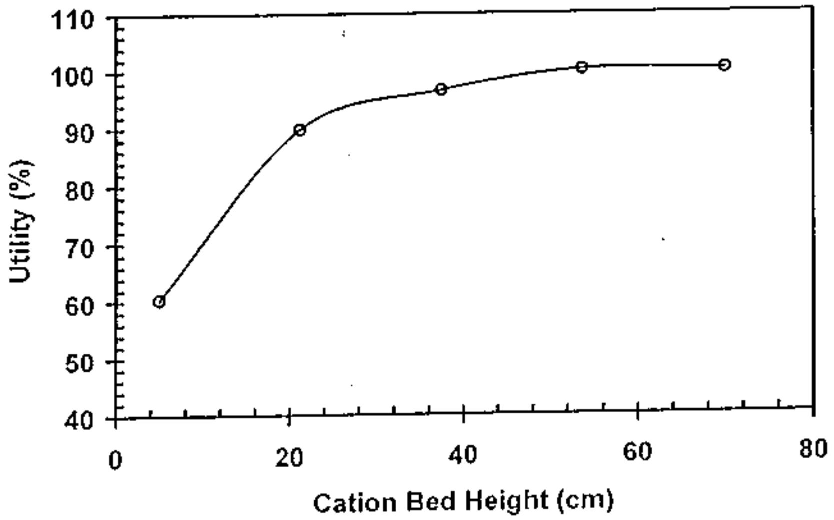


Figure (3.2) Effect of bed depth on percentage of utilized ion exchange capacity

3.2 Experimental Investigation Study

The present experimental investigation study includes in the first stage production of deionized water from salt solution in order to study the most affecting parameters on ion exchanger performance whereas the second investigating study was carried out on the production of deionized water from wastewater of Al-Zawraa Company. Removal of toxic heavy metals and concentrating them in small volumes using 4 % HCl solution as a regenerant was the third experimental investigation. Studying the behavior of ionic exchangers was carried out at different concentration of Cu^{+2} ions, different TDS, different flow rates, and different ion exchanger heights (cation and anion). Precipitation of ions of toxic heavy metals generated from regeneration was studied by adding 20 % CaO aqueous solution. These toxic heavy metals are precipitated as hydroxide when pH rises from 8 to 10. The operating conditions using aqueous solutions containing NaCl, $CuSO_4$ are shown below. Selecting of salts of two kinds of ions Cu^{+2} and Na^{+} are recommended to study their influence on each other. This will give an idea about the behavior of the ion exchangers towards monovalent and divalent ions.

Operating Conditions:
Cu^{+2} concentration ranged between 5 to 25 ppm categorized as X_1
TDS as NaCl and $CuSO_4$ ranged between 250 and 1250 ppm categorized as X_2
Flow rate ranged between 0.38 to 5.34 liter/h categorized as X_3
Bed height of cation ranged between 5 to 70 cm categorized as X_4
Bed height of anion ranged between 7.5 to 105 cm

The concentration of Cu^{+2}, which is actually the major pollutant in wastewater of the electronic printed circuit board in Al-Zawraa Company, was selected within this range from preliminary tests carried out on wastewater of

Al-Zawraa Company. The makeup solution were prepared by weighing NaCl and $CuSO_4$ in sensitive digital devices and added to accurate volumes of deionized water of 10 µs/cm conductivity. The experiments continued until breakthrough point that already decided to be at 40 µs/cm conductivity, was detected. The average conductivity of the total resulted deionized water from each run was ranged between 10 to 15 µs/cm or of TDS content between 5 to 7.5 ppm. Even though, the run was continued until full exhaustion point was detected where output concentration equals to input concentration.

Testing in these experiments was characterized on periodical measurements of electric conductivity of the effluent stream. Each sample was measured three times and then the average values were considered. The TDS content in the sample was obtained by dividing the readings of the electric conductivity by two. The accuracy of these results can be assured by establishing a calibration curve by preparing different solutions, containing different concentrations of NaCl and recording the conductivity of these solutions. The conductivity readings from these experiments were found equal to twice the value of TDS. A sample, for instance, containing 7.5 ppm of NaCl has a conductivity of 15 µs/cm as shown in Table (3.7). A sample brought from Al-Zawraa Company was analyzed and found that TDS content was 620 ppm and has conductivity of 1243 µs/cm.

Table (3.7) Different concentration of TDS and their Conductivity readings

Sample No.	NaCl Concentration (ppm)	Conductivity (µs/cm)	Temperature (°C)
1	1500	2970	24 – 25
2	1200	2402	24 – 25
3	900	1793	24 – 25
4	450	912	24 – 25
5	750	1500	24 – 25
6	300	615	24 – 25
7	150	301.7	24 – 25
8	75	155	24 – 25
9	30	62	24 – 25

10	15	30.5	24 – 25
11	7.5	15.0	24 – 25
12	1.5	3.0	24 – 25
13	0.75	1.5	24 – 25

Sampling from the final pit at Al-Zawraa Company was with drawing through two months. Sampling analysis was carried out at Ib-Sina Company as found in Table (3.8) below.

Table (3.8) analysis of wastewater of Electronic Printed Circuit in Al-Zawraa Company

Kind of Test	Result	Allowable Limits (Disposal into River)
PH	6.2 – 7.4	10 – 10.6
TDS, ppm	600 – 932	1200
TSS, ppm	0 – 16	<30
SO_4^{-2} ppm	295 – 400	<500
NO_3^- ppm	1.3 – 1.5	<50
Cl^- ppm	132 – 216	<400
Cu^{+2} ppm	0.5 – 25	<1
Pb^{++} ppm	0.04 – 0.4	<0.1
Pd^{++} ppm	Nil	
$Fe^{++} + Fe^{+3}$ ppm	0.003	
Mn^{+2} ppm	0.008	
Sn^{+2} ppm	0.01 – 0.1	
Ca^{++} ppm	140	
Mg^{++} ppm	38	
T. H ppm	480 – 608	700
CO_3 ppm	90 – 130	
Na ppm	76 – 115	

Operation Conditions:

TDS values between 317 to 1193 ppm was categorized as X_1

Flow rates between 0.712 to 4.98 liter/hr was categorized as X_2

Bed heights of cation exchanger between 9.4 to 60.6 cm was categorized as X_3

Bed heights of anion exchanger between 14 to 91 cm

Wastewater from Al-Zawraa Company was analyzed and its constituents were found equal to 830 ppm TDS, 8.7 ppm Cu^{++}, 0.33 ppm pb^{++}, 0.071 ppm Sn^{++} and Nil concentration pd^{++}.

If higher TDS values than 830 ppm were needed, NaCl salt is added while deionized water was added to get less values. The experiments are conducted in following sequence: (1) removing the toxic metals by ion exchangers; (2) concentrating them by regeneration solution; (3) precipitating the small volumes of concentrated solution as hydroxides by lime. Monitoring the TDS concentration in deionized water was carried out by measuring the electric conductivity of effluent whereas other ions concentration such as Ca^{++}, Mg^{++}, Cl^-, etc. are measured in Ibn-Sina Company laboratory. Table (3.9) shows the properties of produced deionized water.

The conditions of the 3rd experimental investigation that concerned with the precipitation of toxic heavy metals from regenerated solution are Cu^{++} ion concentrations between 293 to 1707 ppm and pH values between 8.6 to 11.40. The Cu^{++} ion concentrations were selected to be equivalent or higher than Cu^{++} ion concentration found in Al-Zawraa Company wastewater.

Table (3.9) Properties of produced deionized water by treating wastewater of Al Zawraa Company

Kind of Test	Result
PH	7
Na^+	1.32 – 4.6 ppm
Cl^-	2.2 – 7 ppm
Cu^{+2}	0.001 – 0.008 ppm
Pb	Nil
Sn	Nil
Pd	Nil
TDS	Nil (not sensitive)

3.3 Chemicals

The following solutions are used as acidic and alkaline regeneration solutions.

1. 4 % HCl solution for regeneration of cation exchanger.

2. 4% NaOH solution for regeneration of anion exchanger.

3. Strongly acidic cation exchanger, type IR – 120 of mesh size 14-50.
4. Weakly basic anion exchanger Type Lewatit MI of mesh size 20-50.

3.4 Measuring Devices

The following instrumental apparatus were used in this experimental work for determination of concentration of the cation and anion ions of Al-Zawraa wastewater and makeup solution.

1. Flame Atomic Absorption Spectrophotometer, Type Shimadzu AA – 680 G. Japan, was used to determine concentration of Cu^{++}, pb^{++}, Sn^{++}, pd^{++}, Na^{+}, Ca^{++}, Mg^{++}, …etc.
2. Flameless Atomic Absorption Spectrophotometer with graphite furnace atomizer, Type Shimadzu GFA–4B Japan, was used to determine concentration of Cu^{++}, pb^{++}, Sn^{++}, pd^{++}, Na^{+}, Ca^{++}, …etc. when the concentration of the ions is less than 0.01 ppm.
3. pH meter, Type Titro-Processor 686 Metrohm Swiss made.
4. Conductivity meter, Type 83 Copenhagen Denmark made, ranged between 0 to 3000 μs/cm.
5. Conductivity meter, Type Seral Seradest 5600 U. S. A., ranged between 0 to 20 μs/cm.
6. Conductivity meter, Type WTW LF91 Germany, ranged between 0 to 9000 μs/cm.
7. Turbidity meter, Type HACH Company Model 43900 was used to test concentration of $So_4^{=}$, and Cl^{-}.
8. Weight instrument: Type Sartorius Analytic GMBH GOTTINGEN Germany, ranged between 0 – 200 mg.

9. Gas Chromatography, Type Hewlett Packard 438A U. S. A., Flame Ionization

3.5 *Apparatus*

The experimental rig consists of multiple columns for the cation exchanger and anion exchanger incorporated with dosing pumps. A schematic diagram of the apparatus was shown in Fig (3.3). in addition specification of the columns, valves and dosing pumps are listed below:

1. Glass columns of 50-cm long and 2.54 cm diameter.
2. Glass columns of 90-cm long and 2.54 cm diameter.
3. Plastic (TEF) valves to control opening and closing, direction into column to another.
4. Plastic barrels and container of different capacity.
5. Dosing pumps: CFG Prominent Electronic, Type B02–035 Sweden

   Flow rate 0 – 34.92 liter/hr
6. Dosing pumps: Prominent Electronic, Type E 0407 Gmbh Sweden,

   Flow rate 0 – 6.3 liter/hr.
7. Dosing pumps: Alldos, Type M 201–8027 SA Germany.

3.6 *Experimental Procedure*

3.6.1 Conditioning of the Ion Exchangers

Columns C_1 and C_2 are packed with strongly acidic cations exchanger and weakly basic anion exchangers respectively. The two columns were soaked with deionized water with bed volume of two–third of the column volume (i.e., exchanger packed into the column with full mixing of resin during packing the ion exchanger and no bubbles exist between its particles).

3.6.2 Back Washing

Upward washing of the ionic exchangers (columns C_1 and C_2) with 10-bed volume/h for 10 to 15 minutes using industrial water was performed to ensure entertainment of dirt and fine particles from the resin bed. The benefit of this process is to rearrange the particles of the ionic exchanger inside the column (uniformity of packing). Water for back washing was delivered sing pump 1.

3.6.3 Regeneration

3.6.3.1 Regeneration of the Cation Exchanger

Regeneration of the cation exchanger was carried out by 4 % HCl solution. The acid solution was pumped from tank T2 through pump 2 into column C_1 downward with flow rate of 4-bed volume/h. The regeneration was lasted for 75 minutes.

3.6.4 Rinse Process

3.6.4.1 Slow Rinse

Downward 4-bed volume/h of industrial water was pumped into column C1 via pump 1. This step was lasted for 15 minutes.

3.6.4.2 Quick Rinse

Downward 8-bed volume/h of industrial water was pumped into column C1 via pump 1. The quick rinsing step was lasted for 30 minutes.

3.6.5 Regeneration of The anion Exchanger

Downward 4-bed volume/h of 4 % NaOH solution was pumped from tank T3 via pump 3 into column C2. The regeneration of anion exchanger was lasted for 45 minutes.

3.6.6 Rinse Process

3.6.6.1 Slow Rinse

Downward industrial water was pumped into column C1 and column C2 via pump 1 with 4-bed volume/h. The rinsing step was lasted for 15 minutes.

3.6.6.2 Quick Rinse

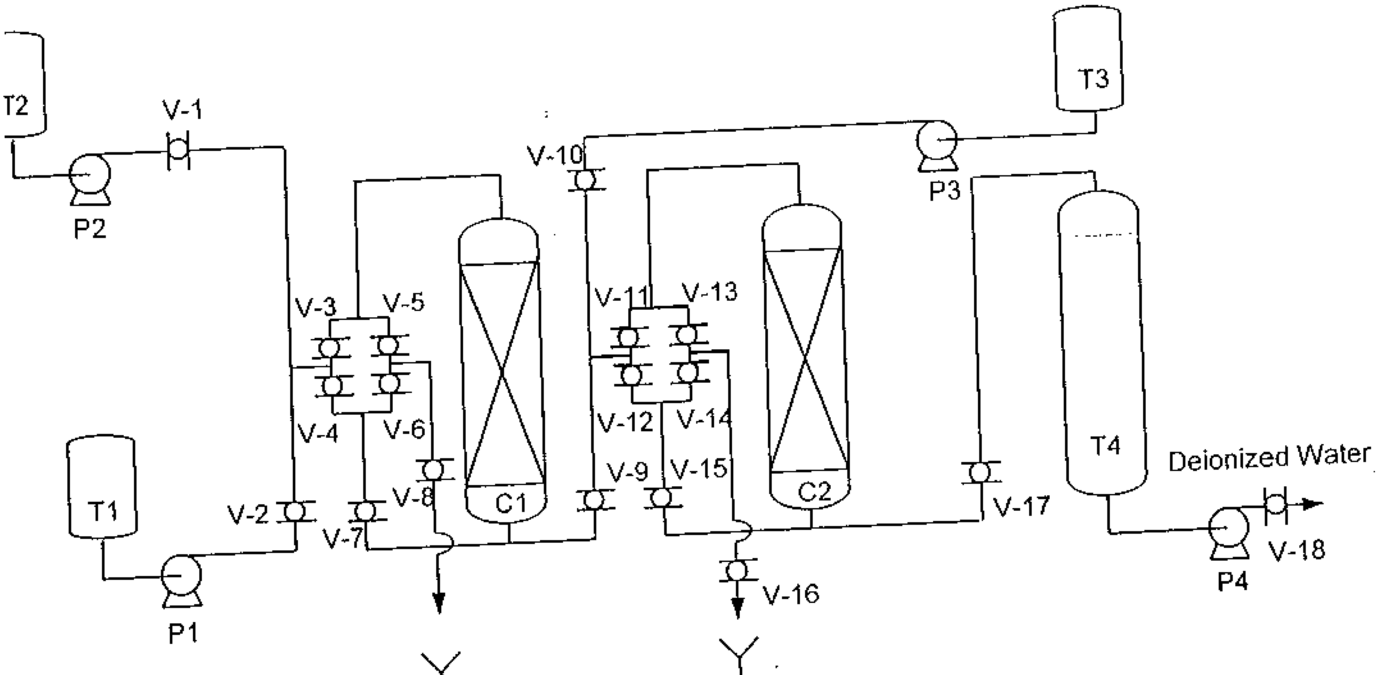
Downward industrial water of 8-bed volume/h was pumped into column C1 and into column C2 via pump No. 1. This step was lasted till a residual conductivity of 40 μs/cm was detected.

3.6.7 Flowing Stage/NaCl-$CuSO_4$ Solution

NaCl/$CuSO_4$ solution is pumped via pump 1 into column C1 (cation exchanger) and into column C2 (anion exchanger) with different feed rates. The concentration feed solution was prepared by dissolving different amounts of NaCl and $CuSO_4$, in which Cu^{+2} ion concentrations were between 5 to 25 ppm. The flowing of solution was kept during the experiment until breakthrough point was detected at conductivity of 40 μs/cm.

3.6.8 Waste Water Flowing Stage

Wastewater was pumped via pump 1 through column C1 (cation exchanger) and column C2 (anion exchanger) with different feed rates. The flowing of solution was kept until breakthrough point was detected at conductivity of 40 μs/cm.



Equipment List	
Item	Description
T1	Wastewater Tank
T2	HCl (4 %) Tank
T3	NaOH (4 %) Tank
T4	Deionized Water Tank
C1	Cation Exchanger Column
C2	Anion Exchanger Column
P	Dosing Pump

Fig (3.3) Flow sheet for Wastewater treatment

Table (3.8) Valves positions in operation, cation backwash and anion backwash, regeneration and rinsing of cation and anion steps

	Valve No.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18
1	Operation	C	O	O	C	C	C	C	C	O	C	O	C	C	C	C	C	O	O
2	Cation Back washing	C	O	C	O	O	C	O	O	C	C	C	C	C	C	C	C	C	C
3	Anion Back washing	C	O	O	C	C	C	C	C	O	C	C	O	O	C	O	O	C	C
4	Cation Regeneration	O	C	O	C	C	O	O	O	C	C	C	C	C	C	C	C	C	C
5	Cation Rinsing	C	O	O	C	C	O	O	O	C	C	C	C	C	C	C	C	C	C
6	Anion Regeneration	C	C	C	C	C	C	C	C	C	O	O	C	C	O	O	O	C	C
7	Anion Rinsing	C	O	O	C	C	C	C	C	O	C	O	C	C	O	O	O	C	C

* C: closed, O: open

3.7 Experimental Design

In general, the purpose of an experimental design is to find useful relationships between controllable variables and observed response. The simplest experiment is a comparison of two things that differ in just one attribute. This sort of experiment obviously has a practical value, but it offers little insight. To gain such insight, more variables must or have to be considered a more sophisticated approach. This classical approach to experimentation is to study one variable at a time, varying its level over a certain range, while holding all other variables constant and observing the effect on the response variable. From these observations, judgement is commenced whether there is a quantitative relationship between variable and response and what the form is, linear, quadratic, etc. Having established such a relation, the effect of other variables would examine in the same manner. This approach is not effective, however, for one thing, it is in efficient, requiring a separate set of observations for each variable and using each observation just once. It is also incapable of detecting interactions. Variables acting together may have a greater or smaller effect than individual variables acting alone. A response surface can be most efficient fitted if proper attention is given to the choice of experimental design (Box and George, 1978).

3.7.1 Principle of Experimental Design

Before studying the process, it is necessary to determine the parameters, which have a considerable effect on the system behavior, or the factors that influence the system objective function. Carrying on several experiments to cover the effect of each parameter as well as the interactions between these parameters if they are not independent. The systematic method, which satisfies the above function with minimum number of experiments, is called "Experimental Design". The application of the experimental design for planing the experiments required to examine the system, will extract the information from pre–existing data by using a statistical method to interpret the results in regular form with the minimum number of observations (Cox, 1958).

This analysis will give description for the system by a correction in order to predict the effect regarding the change of variables on the objective function. Also, the polynomial representation will facilitate the analysis of the system by many techniques, such as optimization of process conditions, required for the maximum value of the objective function (Box and George, 1978; Cox, 1958).

3.7.2 Design for Fitting Second Order Model

An experimental design for fitting a second–order model must have at least three levels for each factor so that the model parameters can be estimated (i.e. variables are usually called factor and the particular value of the variable is called the level). The proper technique for planning a system of more than three variables is "Central Composite Rotatable Design". The total number of treatment combinations is equal to $\left(2^{k}+2k+1\right)$, where k is the number of variables, plus additional further treatments to take the lack of fit and experimental error into account.

3.7.3 Central Composite Rotatable Design

This designs consists of 2^k fractional (i.e. coded to the usual ± 1 notation) augmented by 2k axial points, i.e. $(\pm\alpha, 0, \ldots, 0)$, $(0, \pm\alpha, 0, \ldots, 0)$, $(0, 0, \pm\alpha, \ldots, 0)$, ……, $(0, 0, \ldots, \pm\alpha)$ and center points $(0, 0, 0, \ldots, 0)$. A preliminary step is to set up the relationships between the coded levels and the corresponding real variables. These relationships are as follows:-

$$X_{Coded} = \frac{\left[X_{actual} - X_{center}\right]}{\left[\dfrac{X_{center} - X_{min.}}{\sqrt{k}}\right]} \ldots\ldots\ldots\ldots(3.1)$$

Finally, the number of experiment (n) needed is estimated according to the following equation:

$$n = 2^k + 2k + 1 \ldots\ldots\ldots\ldots(3.2)$$

The value of (n) must be $2^{(k/4)}$ in order to make the design rotatable (Box and George, 1978).

3.7.4 Experimental Trials

The experimental trials using makeup water were designed to produce demineralized water through the following experimental ranges:

(1) Copper concentration between 5 to 25 ppm.

(2) Total dissolved salts (TDS) between 250 to 1250 ppm.

(3) Flow rate of wastewater between 0.38 to 5.34 liter/h.

(4) Bed depth for cation resin between 5 to 70 cm and for anion resin between 7.5 to 105 cm.

The central composite rotatable design of four variables was used and the coded levels are related to real variable values as follows:

$$X_1 = \frac{C_C - 15}{5} \quad \ldots\ldots\ldots\ldots(3.3)$$

$$X_2 = \frac{C_T - 750}{250} \quad \ldots\ldots\ldots\ldots(3.4)$$

$$X_3 = \frac{F - 2.85}{1.23} \quad \ldots\ldots\ldots\ldots(3.5)$$

$$X_4 = \frac{B - 37.5}{16.25} \quad \ldots\ldots\ldots\ldots(3.6)$$

Where C_C is the concentration of copper in ppm, C_T is the total dissolve solid in ppm, F is the feed rate in liter/h, and B is Bed depth of cation resin in cm.

The working ranges of coded and corresponding real variables are listed in Table (3.9). Accordingly there are (29) experiments in a sequence shown in Table (3.11) where the coded values +2, -2, 0 represent the maximum, minimum and average values respectively.

Table (3.9) Coded and corresponding real variables for 1st experimental work

Coded Level	Cu Conc. (ppm)	TDS Conc. (ppm)	Feed Rate (liter/hr)	Bed Depth (cm)
-2	5	250	0.38	5.00
-1	10	500	1.62	21.25
0	15	750	2.85	37.50
1	20	1000	4.08	53.75
2	25	1250	5.34	70.00

Whereas, the experimental work using wastewater from Al–Zawraa Company was designed through the following ranges:

(1) Total dissolved salts (TDS) from 317 to 1193 ppm.

(2) Feed rate of wastewater from 0.712 to 4.98 liter/hr.

(3) Bed depth for cation exchanger from 9.4 to 60.6.

The working ranges of coded and corresponding real variables are listed in Table (3.10), According to experimental design, there are (19) experiments in a sequence shown in Table (3.12) where the coded values +1.732, -1.732, 0 represent the maximum, minimum and average values respectively.

Table (3.10) Coded and corresponding real variables for 2nd experimental work

Coded Level	TDS Conc. (ppm)	Feed Rate (liter/hr)	Bed Depth (cm)
-1.732	317	0.712	9.40
-1	500	1.62	21.25
0	750	2.85	37.5
+1	1000	4.08	53.75
+1.732	1193	4.98	60.60

Table (3.11) Sequence of Experiments According to 1st experimental work (makeup water)

Exp. No.	Coded Variable				Real Variable			
	X_1	X_2	X_3	X_4	Cu^{+2} Conc. (ppm)	TDS Conc. (ppm)	Feed Rate (liter/hr)	Bed Depth (cm)
1	-1	-1	-1	-1	10	500	1.62	21.25
2	1	-1	-1	-1	20	500	1.62	21.25
3	-1	1	-1	-1	10	1000	1.62	21.25
4	1	1	-1	-1	20	1000	1.62	21.25
5	-1	-1	1	-1	10	500	4.08	21.25
6	1	-1	1	-1	20	500	4.08	21.25
7	-1	1	1	-1	10	1000	4.08	21.25
8	1	1	1	-1	20	1000	4.08	21.25
9	-1	-1	-1	1	10	500	1.62	53.75
10	1	-1	-1	1	20	500	1.62	53.75
11	-1	1	-1	1	10	1000	1.62	53.75
12	1	1	-1	1	20	1000	1.62	53.75
13	-1	-1	1	1	10	500	4.08	53.75
14	1	-1	1	1	20	500	4.08	53.75
15	-1	1	1	1	10	1000	4.08	53.75
16	1	1	1	1	20	1000	2.85	37.5
17	-2	0	0	0	5	750	2.85	37.5
18	2	0	0	0	25	750		

19	0	-2	0	0	15	250	2.85	37.5
20	0	2	0	0	15	1250	2.85	37.5
21	0	0	-2	0	15	750	0.38	37.5
22	0	0	2	0	15	750	5.34	37.5
23	0	0	0	-2	15	750	2.85	5.0
24	0	0	0	2	15	750	2.85	70.0
25	0	0	0	0	15	750	2.85	37.5
26	0	0	0	0	15	750	2.85	37.5
27	0	0	0	0	15	750	2.85	37.5
28	0	0	0	0	15	750	2.85	37.5

Table (3.12) Sequence of Experiments According to 2nd experimental work (Al-Zawraa wastewater)

Exp. No.	Coded Variable			Real Variable		
	X_1	X_2	X_3	TDS Conc. (ppm)	Feed Rate (liter/hr)	Bed Depth (cm)
1	1	1	1	1000	4.08	53.75
2	1	1	-1	1000	4.08	21.25
3	1	-1	-1	1000	1.62	21.25
4	-1	1	-1	500	4.08	21.25
5	-1	-1	1	500	1.62	53.75
6	1	-1	1	1000	1.62	53.75
7	-1	1	1	500	4.08	53.75
8	-1	-1	-1	500	1.62	21.25
9	-1.732	0	0	317	2.85	37.5
10	0	-1.732	0	750	0.712	37.5
11	0	0	-1.732	750	2.85	9.355
12	1.732	0	0	1193	2.85	37.5
13	0	1.732	0	750	4.98	37.5
14	0	0	1.732	750	2.85	60.64
15	0	0	0	750	2.85	37.5
16	0	0	0	750	2.85	37.5
17	0	0	0	750	2.85	37.5
18	0	0	0	750	2.85	37.5
19	0	0	0	750	2.85	37.5

Also the experimental study was devoted to test the effect of process variables on precipitation of heavy metals ions especially for copper ions from concentrated solution after regeneration. The experiments were designed in the following ranges: -

(1) Copper concentration between 293 to 1500 ppm.

(2) pH value between 8.6 to 11.4.

The working ranges of coded and corresponding real variables are listed in Table (3.13). According to experimental design, there are (12) experiments in a sequence shown in Table (3.14) where the coded values +1.414, -1.414, 0 represent the maximum, minimum and average values respectively.

Table (3.13) Working range of coded and corresponding real variables

Coded Level	Copper Concentration (ppm)	pH
-1.414	293	08.586
-1	500	09.000
0	1000	10.000
1	1500	11.000
1.414	1707	11.414

Table (3.14) Sequence of experiments according to central composite design

Exp No.	Coded Variable		Real Variable		Cu (ppm)
	X$_1$	X$_2$	Copper Conc. (ppm)	PH	
1	-1	-1	500	9	0.66
2	-1	1	500	11	0.50
3	1	-1	1500	9	0.10
4	1	1	1500	11	0.50
5	0	0	1000	10	0.30
6	0	0	1000	10	0.35
7	0	0	1000	10	0.25
8	0	0	1000	10	0.27
9	1.414	0	1707	10	0.40
10	-1.414	0	293	10	0.75
11	0	1.414	1000	11.414	0.38
12	0	-1.414	1000	8.586	0.2

Chapter
4

Chapter Four

Results & Discussion

Chapter Four
Results and Discussion

4.1 Introduction

Makeup aqueous solutions were used to study the effect of most affecting parameters (i.e. copper concentration, total dissolved solids concentration, feed rate and bed depth) on productivity of deionized water containing. In addition, production of deionized water from Al–Zawraa company wastewaters using ion exchangers of two types were used in terms of total dissolved solids concentration, feed rate and bed depth. These variables had been correlated with adsorption capacity, and breakthrough time by second orders polynomials.

4.2 Adsorption Capacity and breakthrough Time

Tables (4.1), and (4.2) listed the experimental adsorption capacity, and break through time of treated water for the first experimental investigation respectively. Where, Tables (4.3) and (4.4) listed the experimental adsorption capacity, break through time of treated water for the second experimental investigation respectively.

4.3 Response Surface Methodology

Response surface methodology (RSM) is a collection of mathematical and statistical techniques useful for analyzing problems where several independent variables influence a dependant variable or response and the goal is to optimize this response. It is assumed that the independent variables are continuous and controllable by the experimenter with negligible error.

In most RSM problems, the form of the relationship between the response and the independent variables is unknown. Usually, a second order polynomial in some region of the independent variables is employed.

$$Y = B_o + \sum_{i=1}^{k} B_i X_i + \sum_{i=1}^{k} B_{ii} X_i^2 + \sum_{i}\sum_{j} B_{ij} X_i X_j + \varepsilon$$

If the fitted surface is an adequate approximation of Y then analysis of the fitted surface will be approximately to analysis of the actual system.

The model parameters can be estimated most effectively if proper experimental designs are used, customarily, method of least squares is used to estimate the parameters in the approximating polynomial.

Table (4.1) Adsorption Capacity for the ion exchanger, 1st experimental investigation (makeup water)

Exp. No.	Coded Variable				Real Variable				Exp. ml/ml Wet Resin	Pred. ml/ml wet Resin
	X1	X2	X3	X4	Copper Conc. ppm	TDS ppm	Flow Rate l./hr	Bed Depth cm		
1	-1	-1	-1	-1	10	500	1.62	21.25	32.25	31.75
2	1	-1	-1	-1	20	500	1.62	21.25	31.5	31.4
3	-1	1	-1	-1	10	1000	1.62	21.25	32.82	32.9
4	1	1	-1	-1	20	1000	1.62	21.25	31.8	31.77
5	-1	-1	1	-1	10	500	4.08	21.25	20.17	21.33
6	1	-1	1	-1	20	500	4.08	21.25	21.76	21.7
7	-1	1	1	-1	10	1000	4.08	21.25	18.13	18.36
8	1	1	1	-1	20	1000	4.08	21.25	17.45	17.95
9	-1	-1	-1	1	10	500	1.62	53.75	34.36	34.43
10	1	-1	-1	1	20	500	1.62	53.75	34.24	34.09
11	-1	1	-1	1	10	1000	1.62	53.75	34.24	34.37
12	1	1	-1	1	20	1000	1.62	53.75	33.83	33.24
13	-1	-1	1	1	10	500	4.08	53.75	35.9	36
14	1	-1	1	1	20	500	4.08	53.75	35.9	36.39
15	-1	1	1	1	10	1000	4.08	53.75	31.14	31.81
16	1	1	1	1	20	1000	4.08	53.75	30.84	31.41
17	-2	0	0	0	5	750	2.85	37.5	34.63	33.99
18	2	0	0	0	25	750	2.85	37.5	33.23	33.23
19	0	-2	0	0	15	250	2.85	37.5	32.8	32.62
20	0	2	0	0	15	1250	2.85	37.5	29.25	28.79
21	0	0	-2	0	15	750	0.38	37.5	33.9	34.77
22	0	0	2	0	15	750	5.34	37.5	24.03	22.52
23	0	0	0	-2	15	750	2.85	5.0	19.0	18.69
24	0	0	0	2	15	750	2.85	70.0	35.16	34.84
25	0	0	0	0	15	750	2.85	37.5	34.74	34.39
26	0	0	0	0	15	750	2.85	37.5	34.88	34.39
27	0	0	0	0	15	750	2.85	37.5	34.2	34.39
28	0	0	0	0	15	750	2.85	37.5	33.75	34.39

Table (4.2) Experimental breakthrough time for the ion exchanger, 1st experimental investigation (makeup water)

Exp. No.	Coded Variable				Real Variable				Exp. Breakthro. Time (h)	Pred. Breakthro. Time (h)
	X1	X2	X3	X4	Copper Conc. ppm	TDS ppm	Flow Rate l./hr	Bed Depth cm		
1	-1	-1	-1	-1	10	500	1.62	21.25	10.75	15.18
2	1	-1	-1	-1	20	500	1.62	21.25	10.50	15.18
3	-1	1	-1	-1	10	1000	1.62	21.25	5.47	11.42
4	1	1	-1	-1	20	1000	1.62	21.25	5.30	7.73
5	-1	-1	1	-1	10	500	4.08	21.25	2.67	3.55
6	1	-1	1	-1	20	500	4.08	21.25	2.88	0.14
7	-1	1	1	-1	10	1000	4.08	21.25	1.20	-0.35
8	1	1	1	-1	20	1000	4.08	21.25	1.15	0.297
9	-1	-1	-1	1	10	500	1.62	53.75	28.9	34.47
10	1	-1	-1	1	20	500	1.62	53.75	28.8	30.84
11	-1	1	-1	1	10	1000	1.62	53.75	14.4	17.63
12	1	1	-1	1	20	1000	1.62	53.75	14.22	18.06
13	-1	-1	1	1	10	500	4.08	53.75	12.00	10.06
14	1	-1	1	1	20	500	4.08	53.75	12.00	10.77
15	-1	1	1	1	10	1000	4.08	53.75	5.20	0.81
16	1	1	1	1	20	1000	4.08	53.75	5.10	5.59
17	-2	0	0	0	5	750	2.85	37.5	7.69	6.42
18	2	0	0	0	25	750	2.85	37.5	7.38	3.45
19	0	-2	0	0	15	250	2.85	37.5	21.87	18.62
20	0	2	0	0	15	1250	2.85	37.5	3.90	1.93
21	0	0	-2	0	15	750	0.38	37.5	56.50	43.02
22	0	0	2	0	15	750	5.34	37.5	2.90	11.17
23	0	0	0	-2	15	750	2.85	5	0.57	-3.44
24	0	0	0	2	15	750	2.85	70	14.60	13.39
25	0	0	0	0	15	750	2.85	37.5	7.72	7.64
26	0	0	0	0	15	750	2.85	37.5	7.75	7.64
27	0	0	0	0	15	750	2.85	37.5	7.60	7.64
28	0	0	0	0	15	750	2.85	37.5	7.50	7.64

Table (4.3) Experimental adsorption Capacity for the ion exchanger, 2nd experimental investigation (PCB wastewater)

Exp. No.	Coded Variable			Real Variable			Exp. ml/ml. wet Resin	Pred. ml/ml. wet Resin
	X1	X2	X3	TDS Conc. ppm	Flow Rate (l/h)	Bed Depth (cm)		
1	1	1	1	1000	4.08	53.75	31.6	31.04
2	1	1	-1	1000	4.08	21.25	22.67	23.38
3	1	-1	-1	1000	1.62	21.25	30.57	31.23

4	-1	1	-1	500	4.08	21.25	26.55	26.73
5	-1	-1	1	500	1.62	53.75	32.99	32.2
6	1	-1	1	1000	1.62	53.75	34.64	34.38
7	-1	1	1	500	4.08	53.75	35.18	34.45
8	-1	-1	-1	500	1.62	21.25	28.49	28.98
9	-1.732	0	0	317	2.85	37.5	29.96	30.4
10	0	-1.732	0	750	0.712	37.5	33.56	33.46
11	0	0	-1.732	750	2.85	9.355	26.47	25.26
12	1.732	0	0	1193	2.85	37.5	29.75	29.39
13	0	1.732	0	750	4.98	37.5	28.42	28.6
14	0	0	1.732	750	2.85	60.64	33.37	34.68
15	0	0	0	750	2.85	37.5	30.15	30.46
16	0	0	0	750	2.85	37.5	30.7	30.46
17	0	0	0	750	2.85	37.5	30.6	30.46
18	0	0	0	750	2.85	37.5	30.38	30.46
19	0	0	0	750	2.85	37.5	30.45	30.46

Table (4.4) Experimental breakthrough time for the ion exchanger, 2nd experimental investigation (PCB wastewater)

Exp. No.	Coded Variable			Real Variable			Exp. Breakthro. Time (h)	Pred. Breakthro. Time (h)
	X1	X2	X3	TDS Conc. ppm	Flow Rate (l/h)	Bed Depth (cm)		
1	1	1	1	1000	4.08	53.75	5.272	3.03
2	1	1	-1	1000	4.08	21.25	1.5	1.99
3	1	-1	-1	1000	1.62	21.25	5.09	7.26
4	-1	1	-1	500	4.08	21.25	3.51	3.00
5	-1	-1	1	500	1.62	53.75	27.75	28.34
6	1	-1	1	1000	1.62	53.75	14.56	16.17
7	-1	1	1	500	4.08	53.75	11.75	10.67
8	-1	-1	-1	500	1.62	21.25	9.49	12.82
9	-1.732	0	0	317	2.85	37.5	15.75	14.94
10	0	-1.732	0	750	0.712	37.5	29.85	25.94
11	0	0	-1.732	750	2.85	9.355	1.46	-1.17
12	1.732	0	0	1193	2.85	37.5	4.15	3.52
13	0	1.732	0	750	4.98	37.5	3.60	6.06
14	0	0	1.732	750	2.85	60.64	12	13.18
15	0	0	0	750	2.85	37.5	6.7	6.76
16	0	0	0	750	2.85	37.5	6.82	6.76
17	0	0	0	750	2.85	37.5	6.8	6.76
18	0	0	0	750	2.85	37.5	6.75	6.76
19	0	0	0	750	2.85	37.5	6.76	6.76

4.4 Estimation the Coefficient of the Proposed Models

Using the experimental data shown in Tables (4.1), (4.2), (4.3), and (4.4), the coefficients were estimated using nonlinear regression analysis technique via a programmed software named statistica. The number of iterations was terminated when the proportion of variance accounted for was equal to (0.99139, 0.977, 0.96075, and 0.95018) respectively and the correlation coefficient (R) was equal to (0.99569, 0.93149, 0.98018, and 0.97477) respectively. Correlating the four variables with adsorption capacity, and break through time, the following models were determined:

1st Experimental Investigation (Makeup water)

Adsorption capacity (ml/ml wet resin):

$$Y = 30.56797 - 0.18744X_1 - 0.95567X_2 - 3.06209X_3 + 4.03738X_4$$
$$- 0.19515X_1^2 - 0.92142X_2^2 - 1.43642X_3^2 - 1.90765X_4^2 + 0.19659X_1X_2$$
$$+ 0.18129X_1X_3 + 0.0021X_1X_4 - 1.03167X_2X_3 - 0.304185X_2X_4 + 2.9979X_3X_4$$

Breakthrough time (h):

$$Y = 7.642605 - 0.744315X_1 - 4.172569X_2 - 7.962403X_3 + 4.209096X_4$$
$$- 0.677027X_1^2 + 0.658826X_2^2 + 4.862981X_3^2 - 0.666129X_4^2 + 1.015796X_1X_2$$
$$+ 1.08517X_1X_3 + 1.030796X_1X_4 + 1.900425X_2X_3 - 1.333949X_2X_4$$
$$- 1.259574X_3X_4$$

Correlated the three variables with adsorption capacity, and break through time, of the 2nd experimental investigation, the following models were determined:

2nd Experimental Investigation (PCB wastewater)

Adsorption capacity (ml/ml wet resin):

$$Y = 30.45563 - 0.292948X_1' - 1.399427X_2' + 2.720198X_3' - 0.184704X_1'^2$$
$$+ 0.193326X_2'^2 - 0.162373X_3'^2 - 1.397634X_1'X_2' - 0.015875X_1'X_3' + 1.125632X_2'X_3'$$

Break through time (h):

$$Y = 6.766621 - 3.296712X_1' - 5.737603X_2' + 4.142875X_3' + 0.820419X_1'^2$$
$$+ 3.078727X_2'^2 - 0.254468X_3'^2 + 1.136005X_1'X_2' - 1.656272X_1'X_3' - 1.965275X_2'X_3'$$

Optimum values at maximum capacity, and break through time were determined using pattern move Hooks and Jeeves optimization technique and found equal to:

1st experimental investigation:

Maximum Adsorption Capacity

$X_1 = 21$ ppm, $X_2 = 250$ ppm, $X_3 = 5.09$ l/h and $X_4 = 70$ cm

Maximum Breakthrough Time

$X_1 = 5$ ppm, $X_2 = 250$ ppm, $X_3 = 0.38$ l/h, $X_4 = 70$ cm;

2nd experimental investigation:

Maximum Adsorption Capacity

$X`_1 = 1193$ ppm, $X`_2 = 0.712$ l/h, $X`_3 =60.64$ cm

Maximum Breakthrough Time

$X`_1 = 317$ ppm, $X`_2 = 0.712$ l/h, $X`_3 = 60.64$ cm

4.5 Effect of Studied Variables on the Response Functions of Makeup Aqueous Solution

4.5.1 Effect of Studied Variables on Breakthrough Time

To emphases the effect of each variable on breakthrough time clearly and individually, each variable was studied separately from the other variables as they kept at constant values (i.e. optimum conditions). As shown in Figs (4.1) to (4.4) the effect of studied variables (i.e., copper concentration, total dissolved solids, flow rate and bed depth) on the breakthrough time were investigated. These figures indicates that the breakthrough time of the ion exchanger is decreased with increase in copper concentration, total dissolved solids concentration and flow rate and increased with longer bed height. The reasons for these characteristics are attributed as follows:

1. Various criteria influencing the general performance of ion exchange resin, however, relative selectivity of the resin for various ionic species has been considered into account in this situation since a strong cation resin

operating in the H^+ form will preferentially exchange ions with different selectivity. It seems that earlier breakthrough time starts to occur with higher concentration of copper ions because Cu^{+2} ions start to replace Na^+ ions in the cation exchanger also SO_4^{-2} ions starts to replace Cl^- ions in the anion exchanger due to their higher selectivities that resulted in leak off the resin earlier.

2. With higher concentration of total dissolved solids, the concentration of ions in solution increases that rises the mass transfer rate through the film until it exceeds the diffusion rate through the resin beads. Then diffusion through the resin becomes the controlling factor and the system is said to exhibit particle controlled kinetics, which is slower process. This condition means that the leakage curve is spread out over deeper reaction zone. If this condition is happened it leads to shorter exhaustion time and lower operating capacity.

3. If the flow rate is slow enough, equilibrium is established as the solution reaches a new layer of the resin that means the wavefront is only slightly diffuse as a result of each successive layer of resin being almost completely exhausted before leakage occurs. As flow rate increases, equilibrium is no longer reached and the exhaustion curve is spread out over deeper reaction zone which has a smaller operating capacity and earlier breakthrough point. Furthermore, the curve tends to flatten (i.e., no further tendency to decrease) at higher flow rate. This was attributed that if the flow rate is slow, the diffusion rates through the resin mass are much greater than that through the surrounding film. The film thus controls the rate of exchange, and the process exhibits film controlled kinetics. If the flow rate is increased further, the mass transfer through the film rises until exceeds the diffusion rate through the resin beads. Then, diffusion through the resin becomes the controlling factor, and the system is said to exhibit particle controlled kinetics where no influence might attribute from further increase in flow rate.

4. The operating capacity is defined as the proportion of total capacity used during the exchange process. It can amount to a large or small proportion of the total capacity and depends on a number of process variables including depth of resin bed. It was simply observed that the longer the column, the greater is the operating capacity of the resin and consequently later breakthrough time which is simply attributed to the deeper is the reaction zone.

To study the interaction effects between the studied variables, Figs (4.5) to (4.10) were drawn. The effect of total dissolved solids concentration on the breakthrough time at different copper concentration was studied as represented in Fig (4.5). It shows that increasing the TDS concentration will decrease the break through time as outlined above in studying the effect of TDS concentration on breakthrough time. This observation had come in according to Neretnieks (1976) justification who had previously pointed out that diffusion rate is controlled by the concentration gradient that takes longer contact time to reach adsorption equilibrium and exhaustion point for the case of low values of initial solute concentration. However, at higher concentration of TDS the resin leaks off earlier with lower copper concentration, which indicates reverse action of copper concentration on the breakthrough time (i.e., at coded value -2 corresponding to real value 5 ppm). This can be attributed to pronounce selectivity of Cu^{++} ions with respect to Na^+ ions at higher concentration of Cu^{+2} that resulted in lately breakthrough time in comparison to lower concentration. Besides, $H^+.Na^+$ exchange, uptake of Na^+ ions is faster than $Na^+.Cu^{+2}$ exchange, uptake of Cu^{+2} ions, by several times. That means at higher concentration of TDS (Concentration of NaCl) and lowers concentration of copper ions, the uptake of Cu^{+2} ions might be faster and consequently earlier breakthrough time might occur.

Figure (4.6) demonstrates the interaction effect between flow rate and copper concentration. The breakthrough time decreases with increasing flow rate as previously monitored in Fig (4.3). Also, Fig (4.1) shows earlier breakthrough time with increasing copper concentration. However, same observation has been noticed at the extreme limit of flow rate (i.e., high flow rate) as found in Fig (4.5) that increasing copper concentration has shown lately breakthrough time. Once again, this can be attributed as declared above to the lower concentration of copper ions that resulted in faster uptake of Cu^{+2} ions and earlier breakthrough time.

Figure (4.7) demonstrates the interaction effect between bed depth and copper concentration. It seems that increasing the bed depth whatever copper concentration will increase the breakthrough time. This was attributed as previously outlined that the longer the column, the deeper is the reaction zone, and the greater is the operating capacity of the resin and consequently lately breakthrough time. But, herein it seems that the bed depth has pronounced effect on breakthrough time relative to copper concentration even though the two variables have opposite effect on breakthrough time, but still bed height tends to higher breakthrough time as if there is no influence of copper concentration on the breakthrough time.

Figure (4.8) shows the interaction effect between flow rate and TDS concentration. In this figure it seems that no interaction had been noticed, besides, the effect of both variables had come in according with each other in the direction of decreasing breakthrough time with increasing flow rate and TDS concentration. Figure (4.9) shows the interaction effect between bed depth and TDS concentration. Herein, the concentration of TDS almost counterbalance the effect of bed height on breakthrough time especially at highest concentration of TDS since no significant variations in breakthrough time was observed with increasing bed height.

Figure (4.10) shows the interaction effect between flow rate and bed depth. Both variables tend to affect the breakthrough time in opposite direction but it seems that the flow rate has the most significant effect on breakthrough time in comparison to bed height.

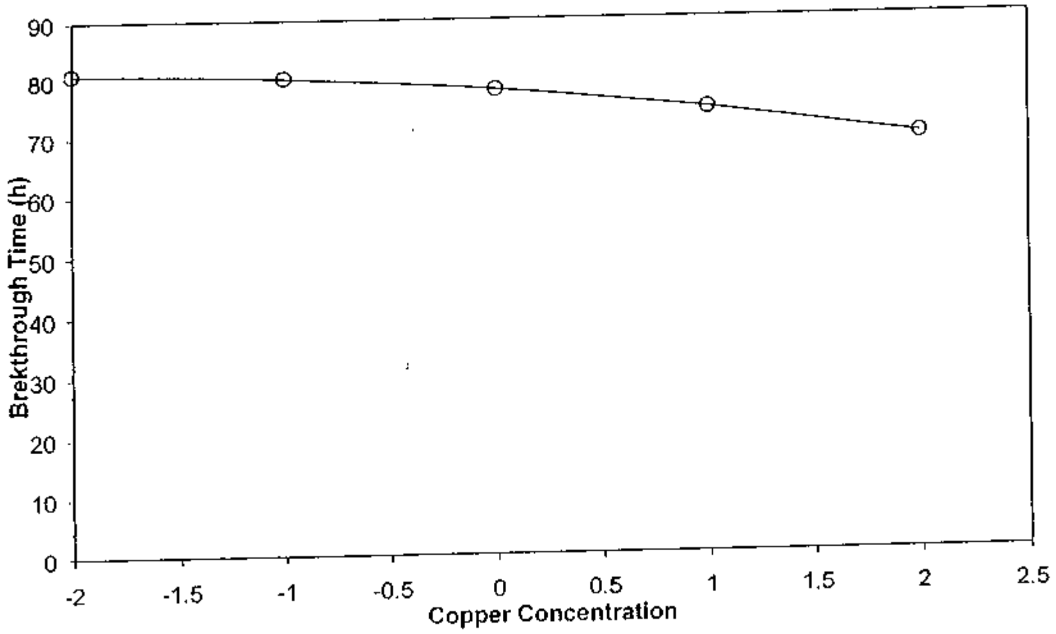


Fig (4.1) Effect of copper concentration on the breakthrough time

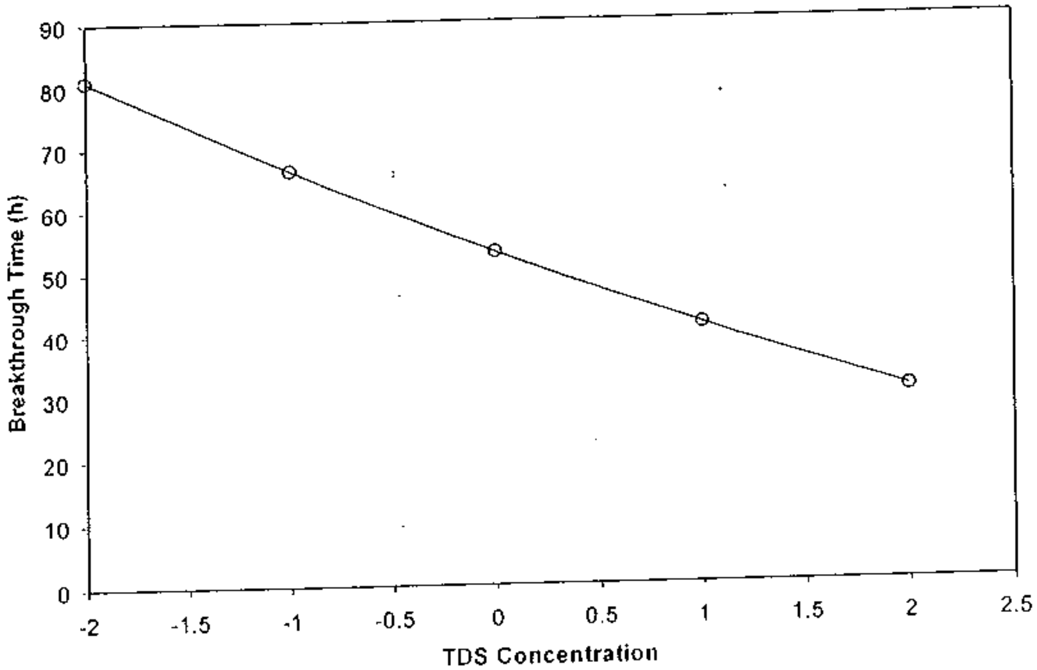


Fig (4.2) Effect of TDS concentration on the breakthrough time

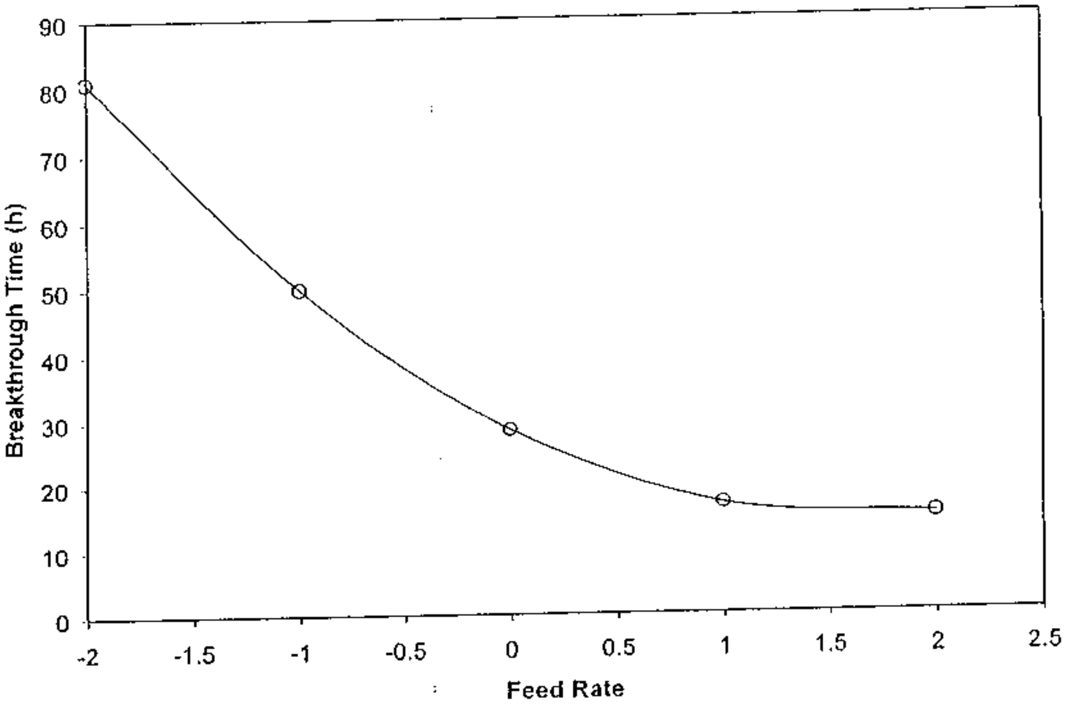


Fig (4.3) Effect of Feed rate on the breakthrough time

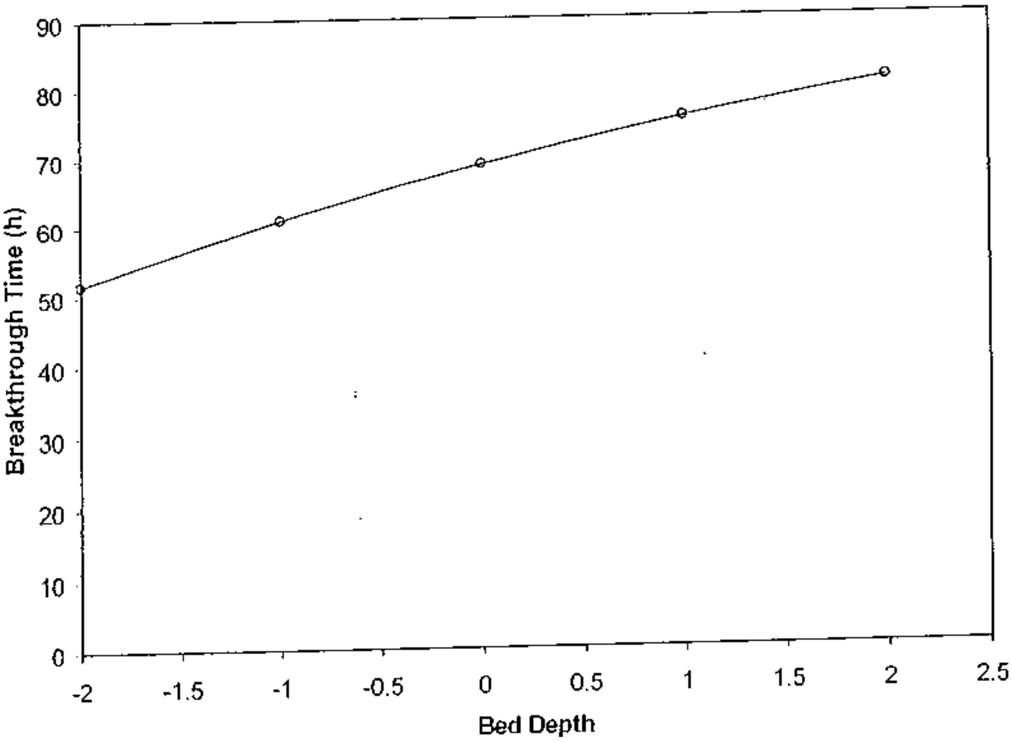


Fig (4.4) Effect of bed depth on the breakthrough time

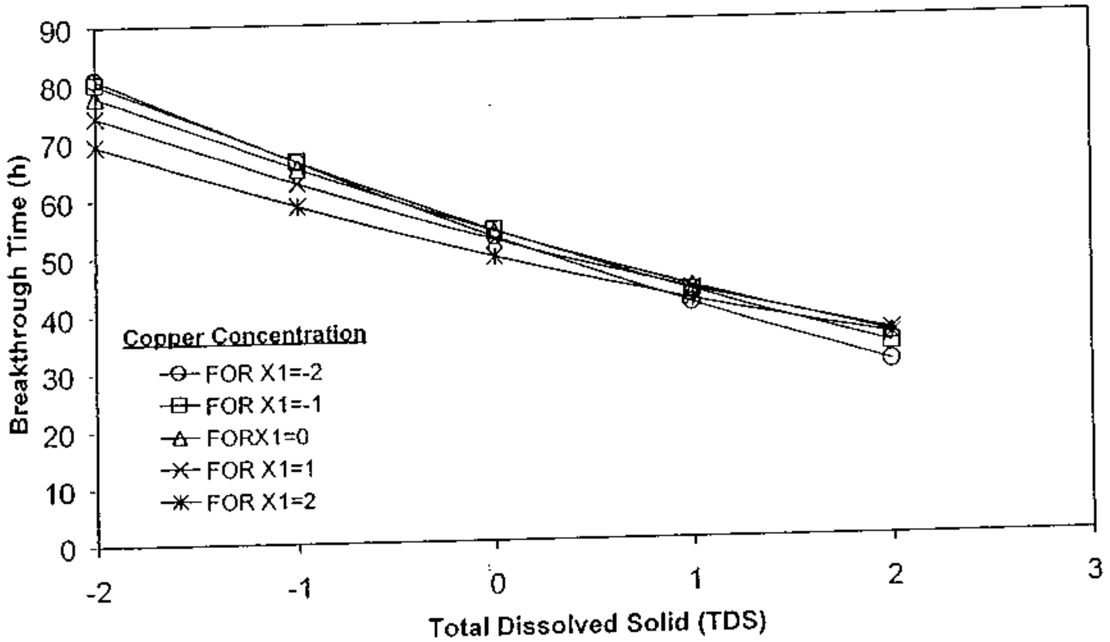


Fig (4.5) Effect of copper concentration and TDS concentration on the breakthrough time

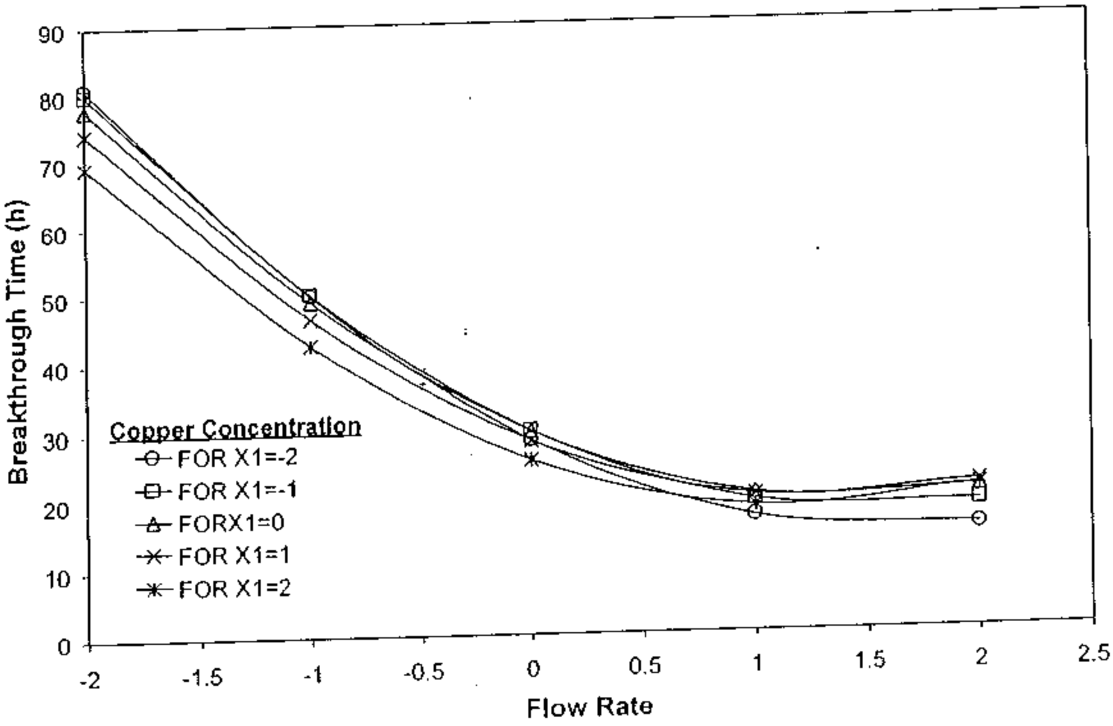


Fig (4.6) Effect of copper concentration and flow rate on the breakthrough time

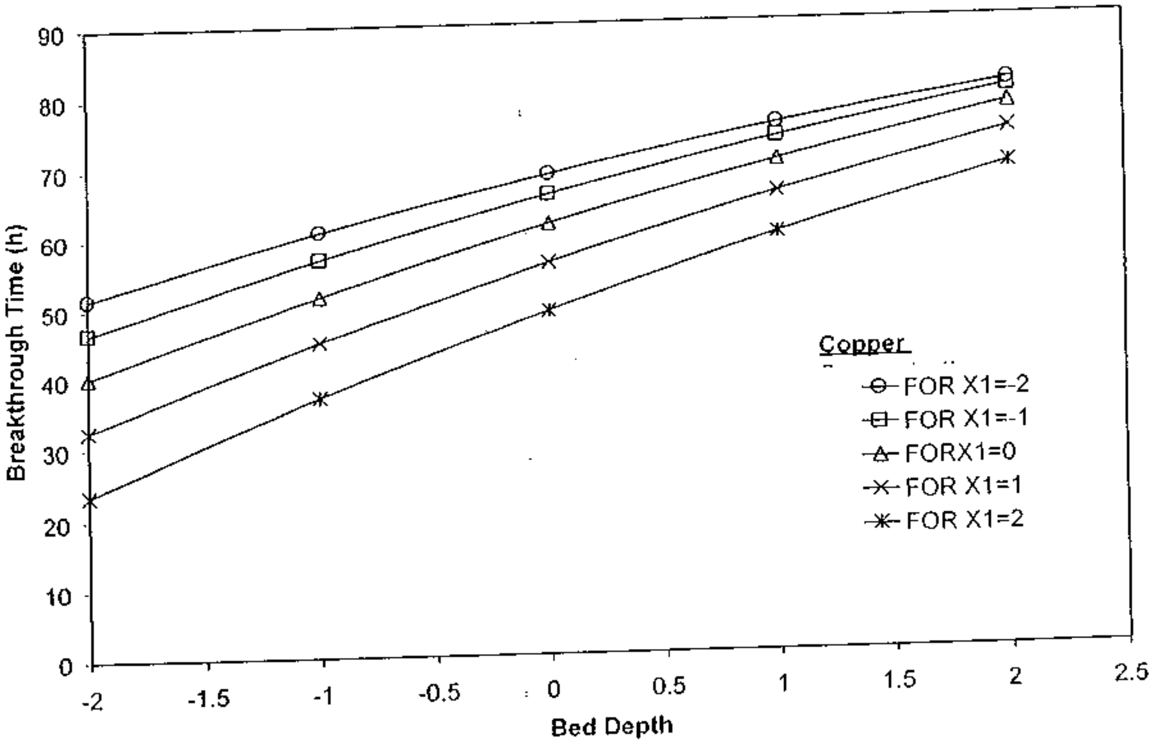


Fig (4.7) Effect of copper concentration and bed depth on the breakthrough time

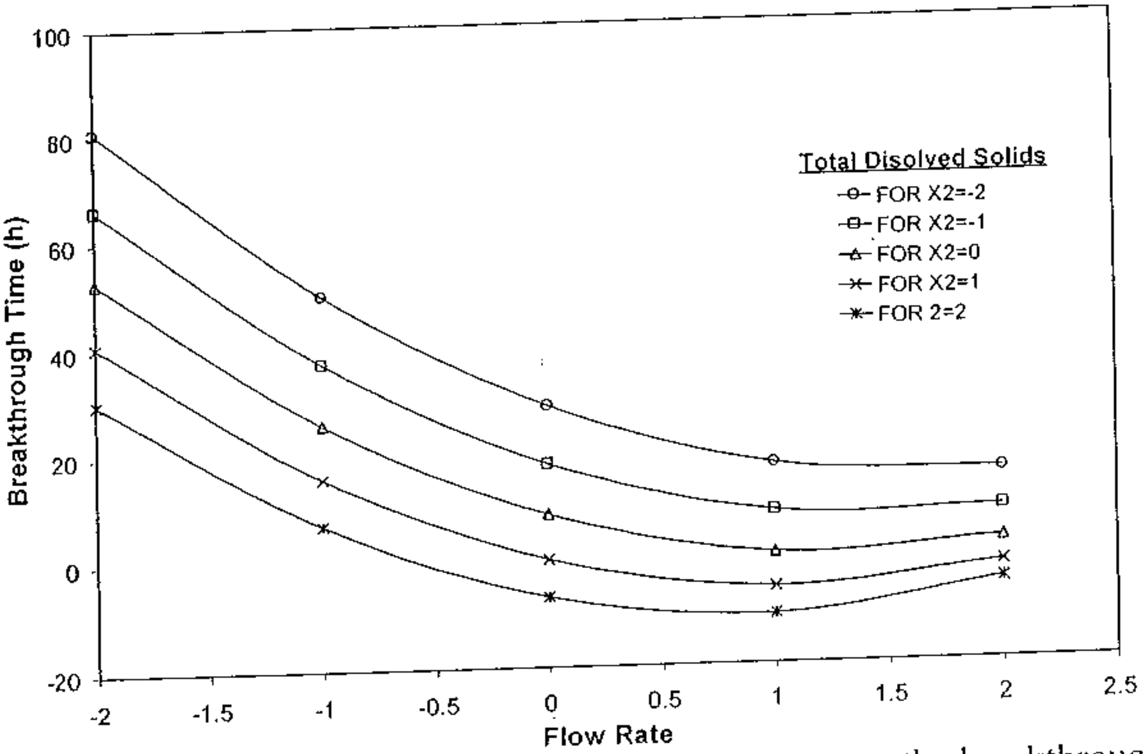


Fig (4.8) Effect of TDS concentration and flow rate on the breakthrough time

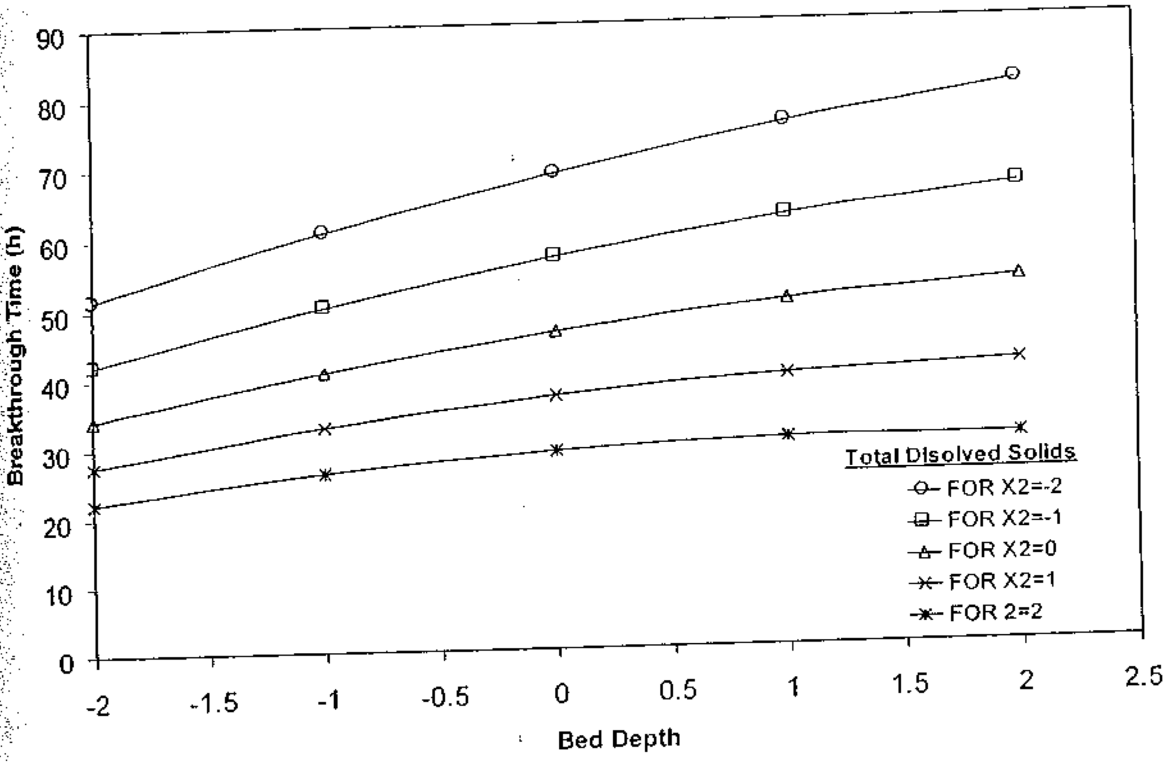


Fig (4.9) Effect of TDS concentration and bed depth on the breakthrough time

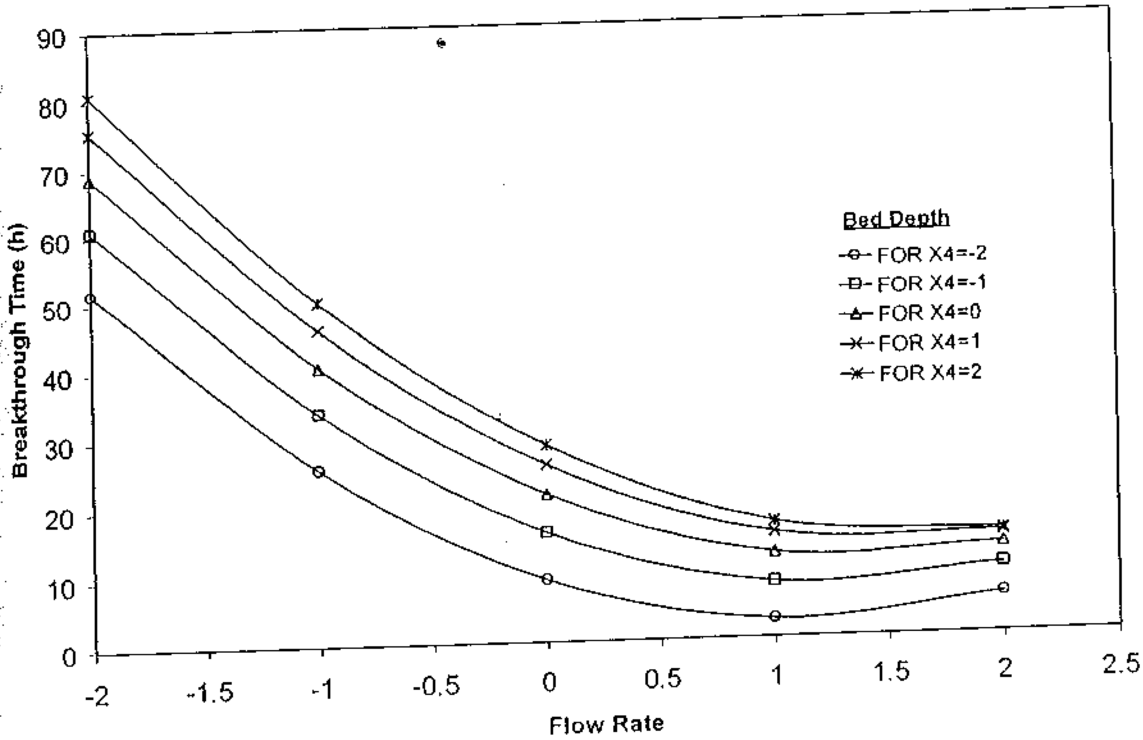


Fig (4.10) Effect of bed depth and flow rate on the breakthrough time

4.5.2 Effect of Studied Variables on Adsorption Capacity

To emphases the effect of each variable on adsorption capacity clearly and individually, each variable was studied separately from the other variables as they kept at constant values (i.e. optimum conditions). As shown in Figs (4.11) to (4.14) the effects of studied variables (i.e., copper concentration, total dissolved solids, flow rate and bed depth) on the adsorption capacity were investigated. These figures indicate that the adsorption capacity of the ion exchanger is increased with copper concentration, flow rate and bed depth and decreased with higher values of TDS.

Fig (4.11) show slight variation of adsorption capacity with copper concentration in comparison with other studied variables. Since adsorption capacity can amount to a large or small proportion of the total capacity, it depends on a number of process variables including ionic concentration. It was simply observed that higher copper concentration lead to greater operating capacity as a result of deeper reaction zone. However, at higher copper concentration, the operating capacity was started to reduce. This can be attributed to pronounce selectivity of Cu^{++} ions in comparison to Na^+ ions at higher concentration of Cu^{+2} that result lesser breakthrough time and consequently lower adsorption capacity. Besides, at lower copper concentration, the $H^+.Na^+$ exchange, uptake of Na^+ ions, is dominate which is faster than $Na^+.Cu^{+2}$ exchange, uptake of Cu^{+2} ions, by several times. This means at higher concentration of copper the uptake of Cu^{+2} ions might be faster that result in lesser breakthrough time and consequently lower adsorption capacity might occur.

Fig (4.12) demonstrate the effect of TDS concentration on adsorption capacity that shows lower adsorption capacity with higher TDS concentration.

This was directly contributed to higher mass transfer rates through the film until it exceeds the diffusion rate through the resin beads and consequently the leakage curve is spread out over deeper reaction zone that means shorter exhaustion time and lower operating capacity.

In Fig (4.13), higher adsorption capacity was noticed with higher flow rates. At slow enough flow rate the wavefront is only slightly diffuse as a result of each successive layer of resin being almost completely exhausted before leakage occurs. As flow rate increases, the exhaustion curve is spread out over deeper reaction zone which has a smaller operating capacity and earlier breakthrough point however it doesn't mean smaller adsorption capacity. Furthermore the curve tends to flatten (i.e., no further tendency to increase) at higher flow rate. Once again, at slow flow rate, the process exhibits film controlled kinetics. If the flow rate is increased further, the diffusion through the resin becomes the controlling factor, and the system is said to exhibit particle controlled kinetics where no influence might attribute from further increase in flow rate.

Fig (4.14) show the effect of bed depth on adsorption capacity. It was simply observed that longer column means greater adsorption capacity, which is simply attributed to the deeper, is the reaction zone. Although, the effect of bed depth upon capacity has not been adequately explained by any existing theory, it is perfectly logical to assume that a minimum bed depth must exist below which leakage will occur immediately.

To study the interaction effects between the studied variables, Figs (4.15) to (4.20) were drawn. The effect of copper concentration on the adsorption capacity at different TDS was studied as represented in Fig (4.15). It shows that at lower TDS concentration, higher adsorption capacity was noticed with higher concentration of copper. This was mirrored at high TDS concentration as the adsorption capacity shows lower value at higher concentration of copper. This can be attributed to pronounce selectivity of Cu^{++} ions with respect to Na^{+} ions at higher concentration of Cu^{+2} that resulted in higher adsorption capacity at lower concentration of TDS. Whereas, at the other extreme limit of TDS concentration, the uptake of Cu^{+2} ions might be faster and consequently lower adsorption capacity might occur with higher copper concentration.

Figure (4.16) demonstrate the interaction effect between flow rate and copper concentration. The adsorption capacity increases with increasing flow rate as previously monitored in Fig (4.13). Also, Fig (4.11) show lower adsorption capacity with increasing copper concentration. However, flow rate has shown positive dependence of greater significance than copper concentration.

Figure (4.17) show the interaction effect between bed depth and copper concentration. It seems that increasing the bed depth whatever copper concentration will increase the adsorption capacity. This was attributed as previously outlined that the longer the column, the deeper is the reaction zone, and the greater is the adsorption capacity of the resin. Although, the two variables have opposite effect on adsorption capacity, but still bed height tends to create higher adsorption capacity as if there is no influence of copper concentration on the breakthrough time.

In Fig (4.18), the interaction effect between flow rate and TDS concentration was monitored. It seems that at lower limit of TDS concentration, the adsorption capacity shows higher values with flow rate. Whereas, at the upper extreme limit of TDS concentration, higher flow rate will show lower adsorption capacity, especially at the upper limit of flow rate at coded value +2.

Figure (4.19) show the interaction effect between bed depth and TDS concentration. Simply, no interaction was observed between TDS concentration and bed depth. Finally, Figure (4.20) shows significant interaction effect between flow rate and bed depth. At longer bed depth (i.e., +2 and +1), higher adsorption capacity was monitored with increasing flow rate. Whereas, at shorter bed depth (i.e., -1 and –2), lower adsorption capacity with increasing flow rate was observed. This behavior was previously pointed out that although the effect of bed depth upon capacity has not been adequately explained by any existing theory, it is logical to assume that a minimum bed depth must exist below which leakage will occur immediately. In other wards, at lower bed depth leakage has occurred earlier that resulted in lower adsorption capacity.

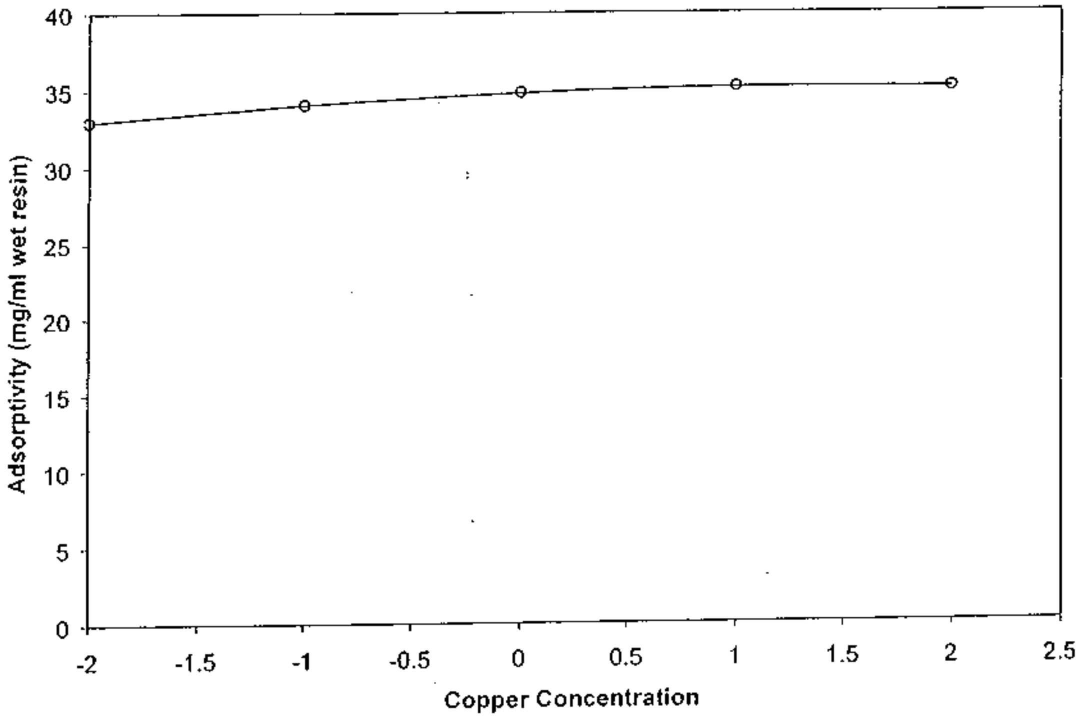


Fig (4.11) Effect of copper concentration on adsorption capacity

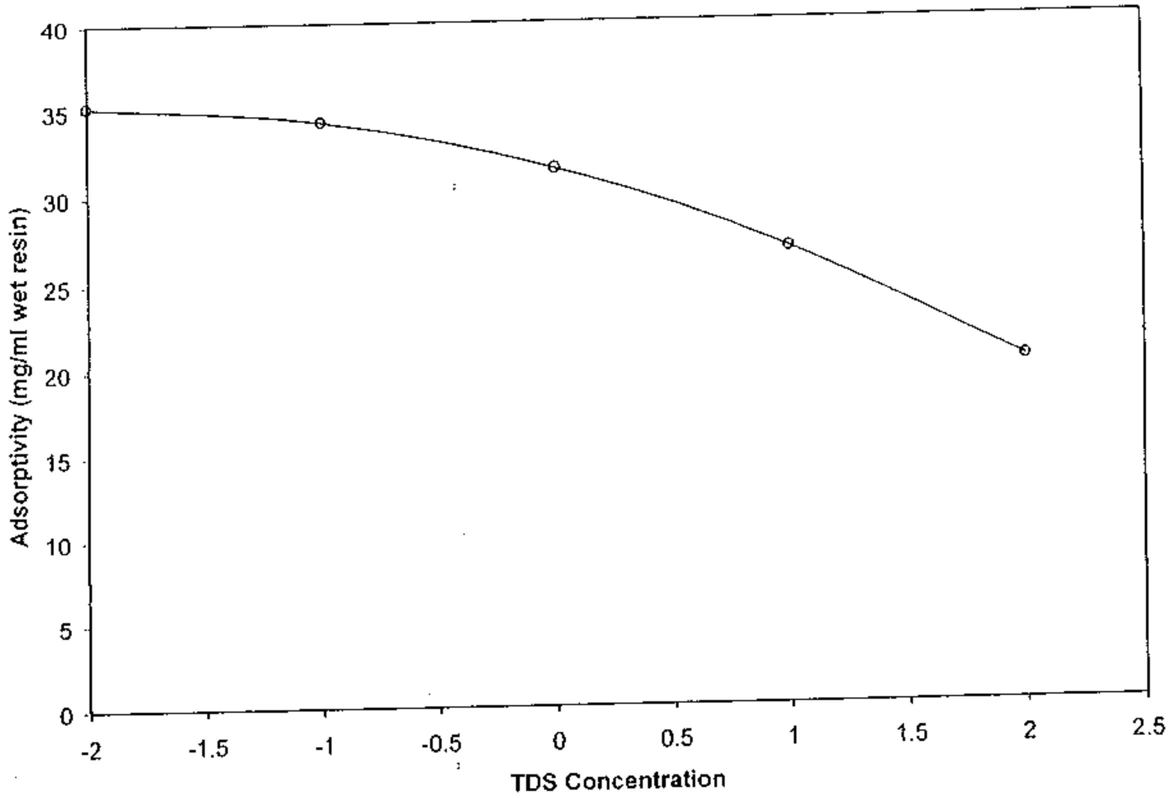


Fig (4.12) Effect of TDS concentration on adsorption capacity

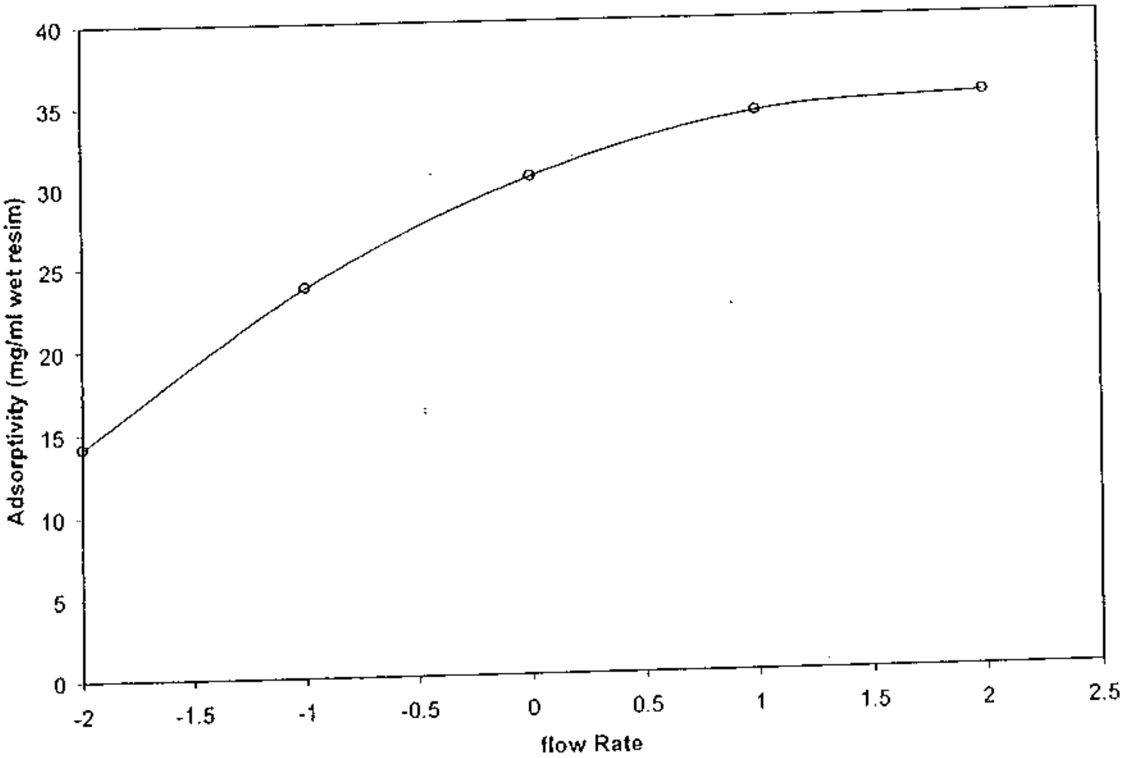


Fig (4.13) Effect of flow rate on adsorption capacity

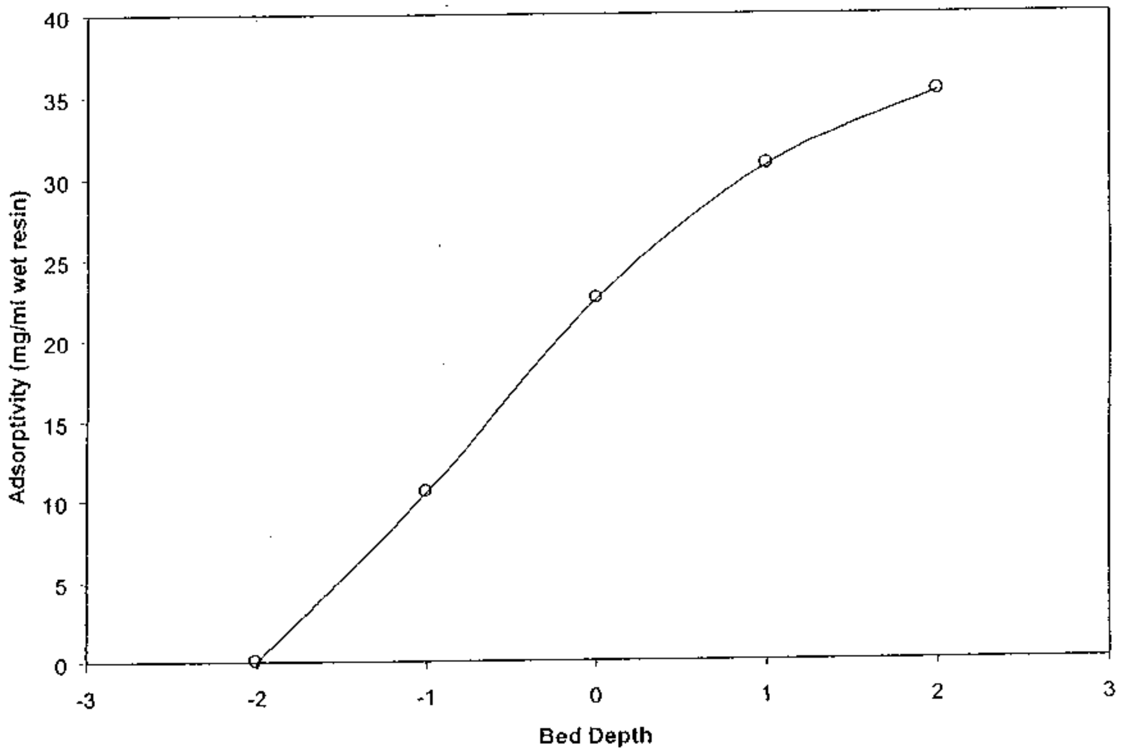


Fig (4.14) Effect of bed depth on adsorption capacity

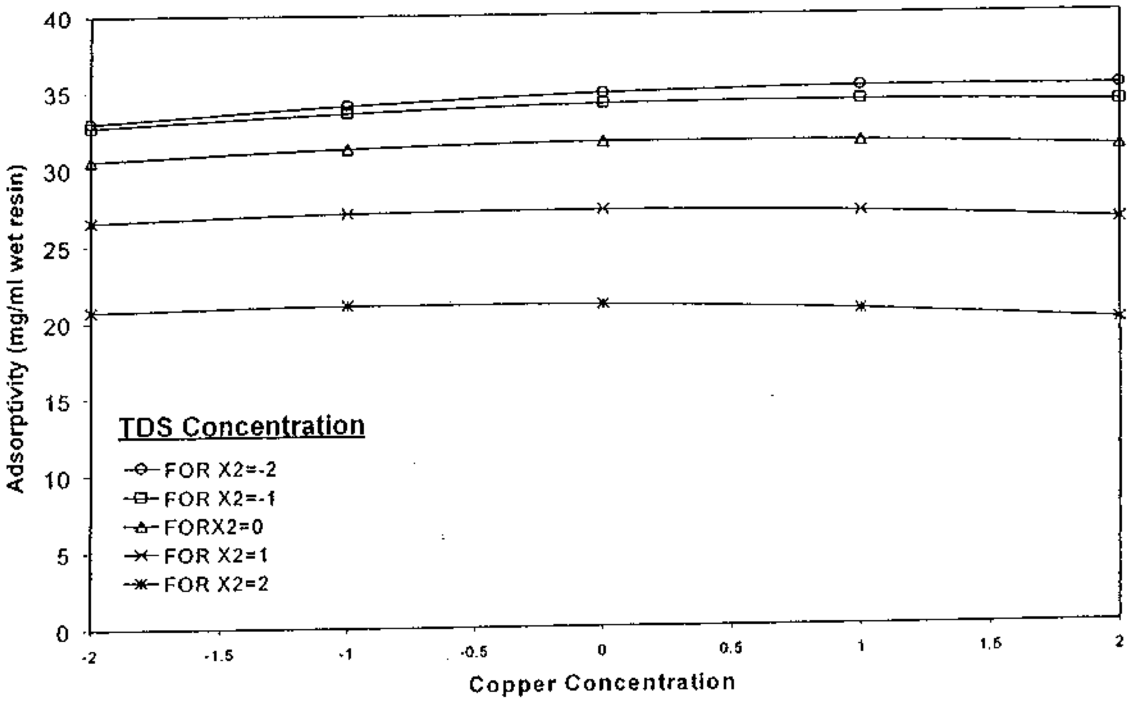


Fig (4.15) Effect of copper concentration and TDS concentration on adsorption capacity

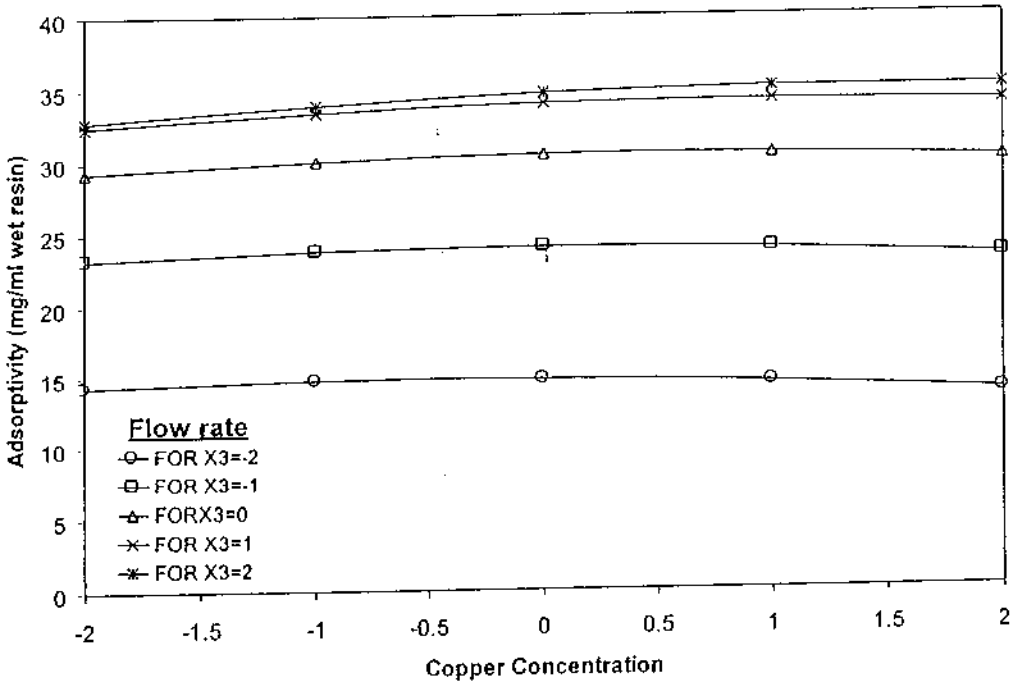


Fig (4.16) Effect of copper concentration and flow rate on adsorption capacity

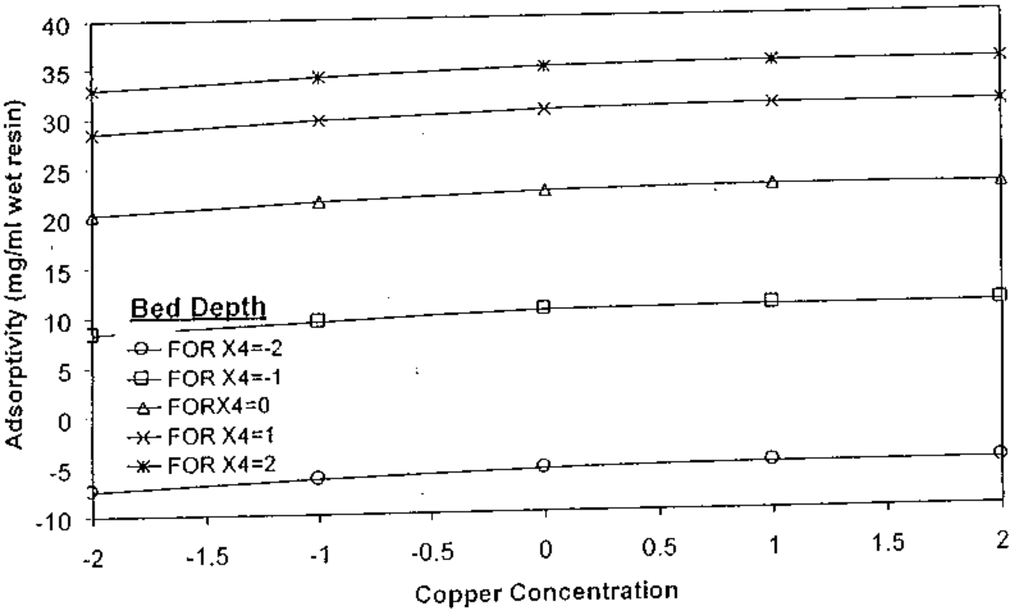


Fig (4.17) Effect of copper concentration and bed depth on adsorption capacity

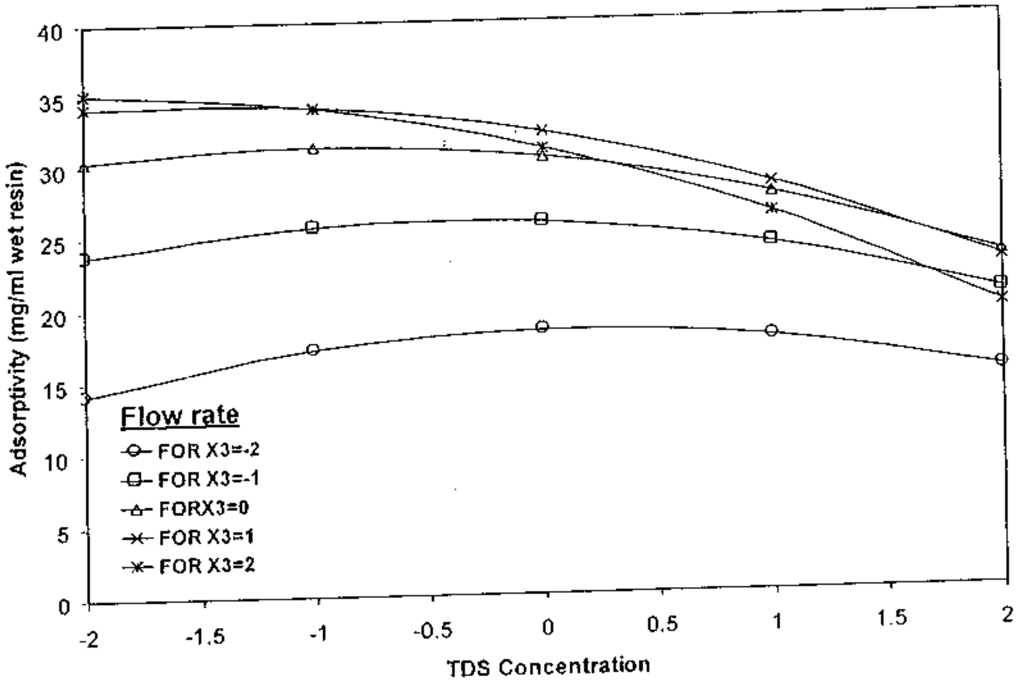


Fig (4.18) Effect of flow rate and TDS concentration on adsorption capacity

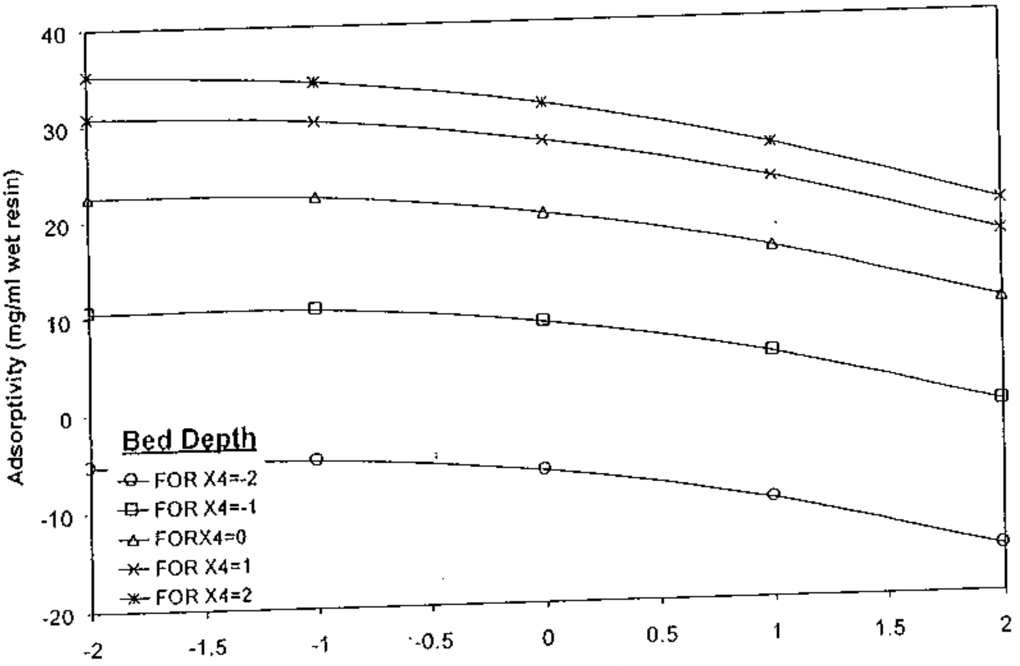


Fig (4.19) Effect of TDS concentration and bed depth on adsorption capacity

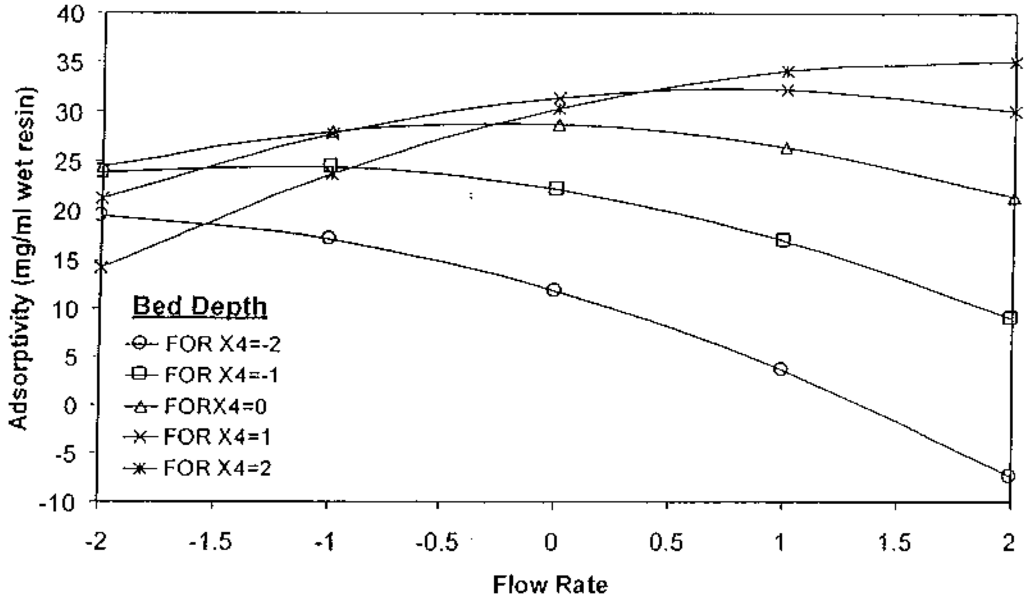


Fig (4.20) Effect of flow rate and bed depth on adsorption capacity

4.6 Effect of Studied Variables on the Response Functions of Al-Zawraa Wastewater

4.6.1 Effect of Studied Variables on Breakthrough Time

Herein, the effect of three variables on the breakthrough time have been considered such as total dissolved solids concentration, flow rate and bed depth. To emphases the effect of each variable on breakthrough time of Al-Zawraa wastewater clearly and individually, each variable was studied separately from the other variables as they kept at constant values (i.e. optimum conditions). As shown in Figs (4.21) to (4.23) the effects of studied variables (i.e., total dissolved solids concentration coded as X_1, flow rate coded as X_2 and bed depth coded as X_3) on the breakthrough time were investigated. These figures indicates that the breakthrough time of the ion exchanger is decreased with increase in total dissolved solids concentration and flow rate and increased with longer bed height. Al-Zawraa wastewater system behaves similar to aqueous makeup solution. The reasons for these characteristics are attributed as previously reasoned for in section 4.5.1 with respect to TDS concentration, flow rate and bed depth.

Same thing is said about the interaction effects between the three studied variables and their effects on breakthrough time in comparison with their respective figures as already outlined in section 4.5.1. Figure (4.24) to (4.26) were drawn to investigate these effects. The effect of total dissolved solids concentration on the breakthrough time at different flow rate was studied in Fig (4.24). While, Fig (4.25) demonstrates the interaction effect between TDS concentration and bed depth. In Fig (4.26), the interaction effect between flow rate and bed depth was monitored.

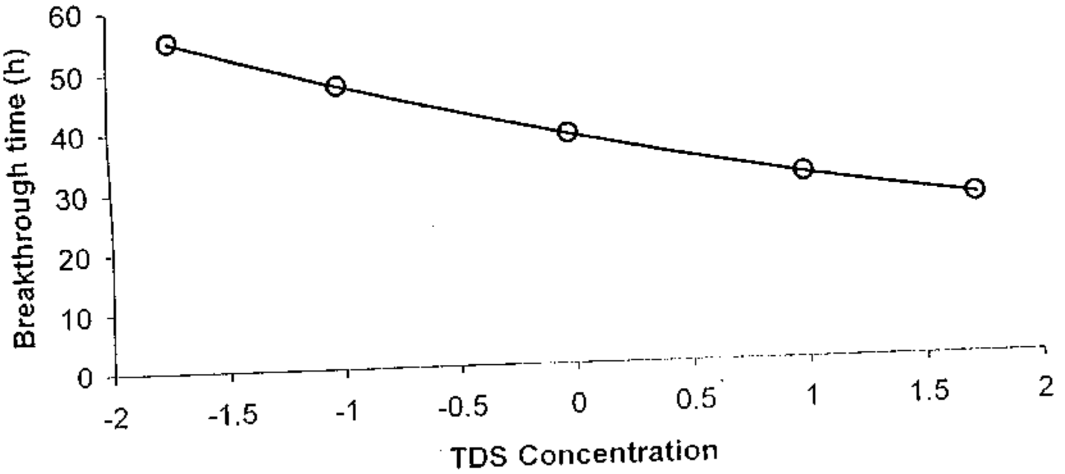


Fig (4.21) Effect of TDS concentration on breakthrough time

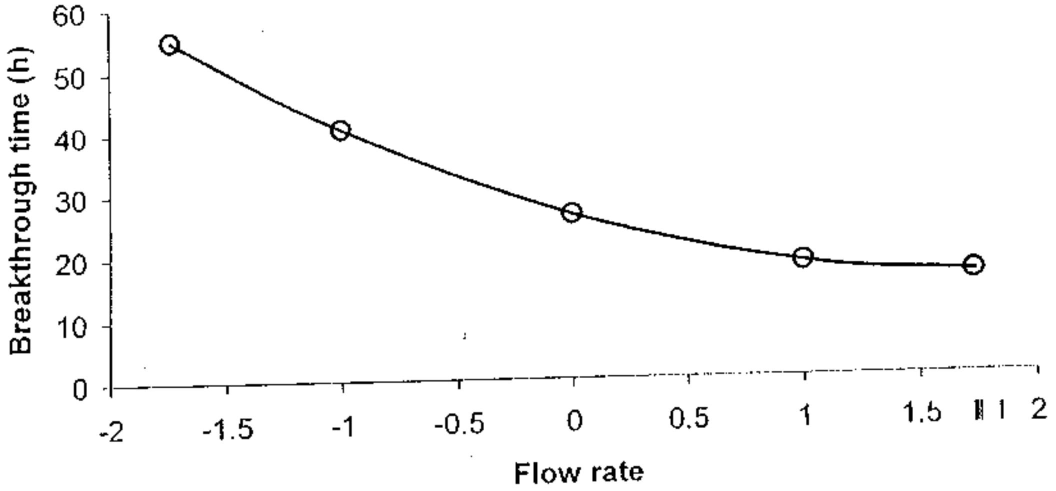


Fig (4.22) Effect of flow rate on breakthrough time

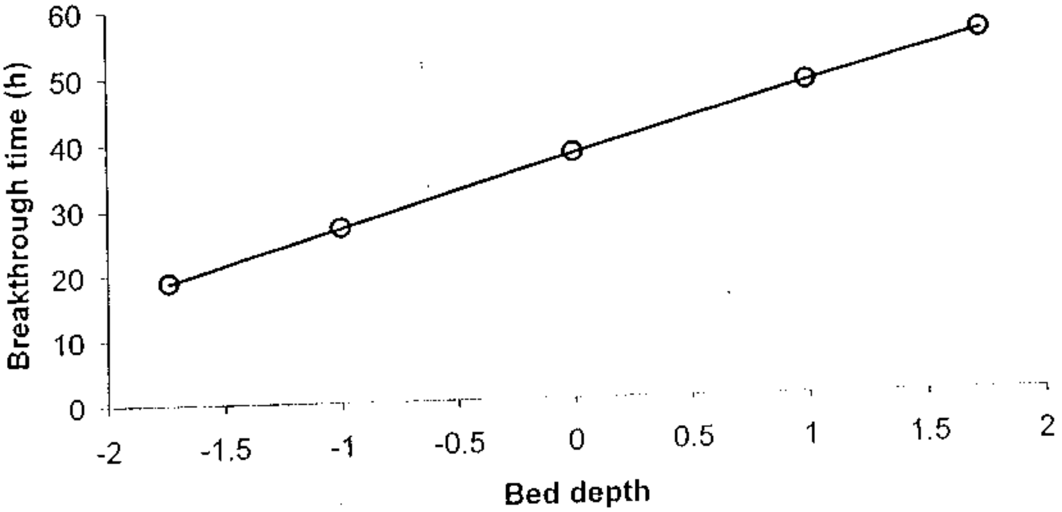


Fig (4.23) Effect of bed depth on breakthrough time

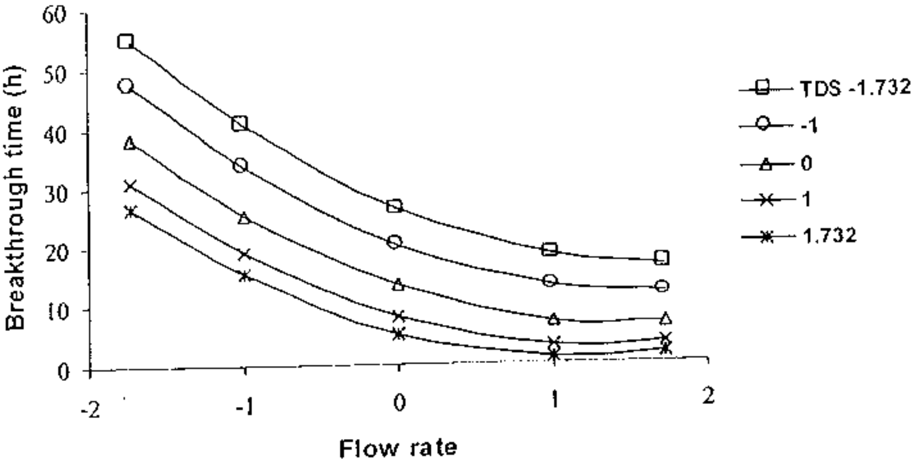


Fig (4.24) Effect of flow rate and bed depth on breakthrough time

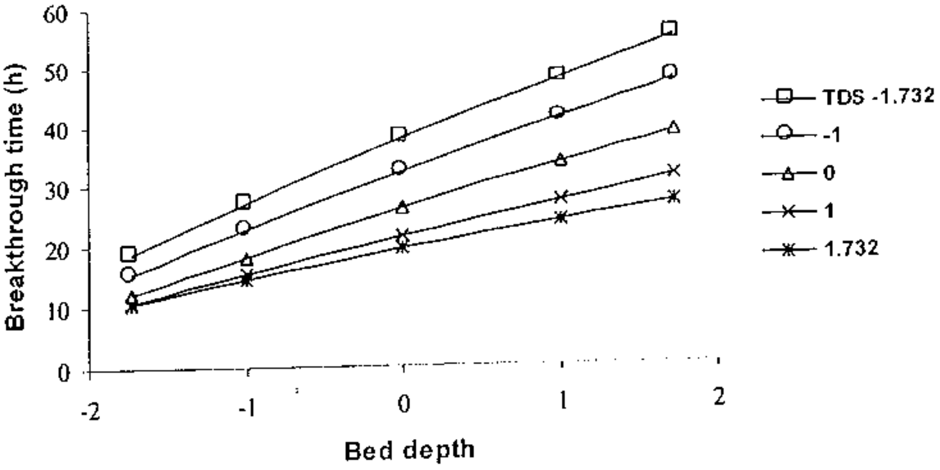


Fig (4.25) Effect of bed depth and TDS concentration on breakthrough time

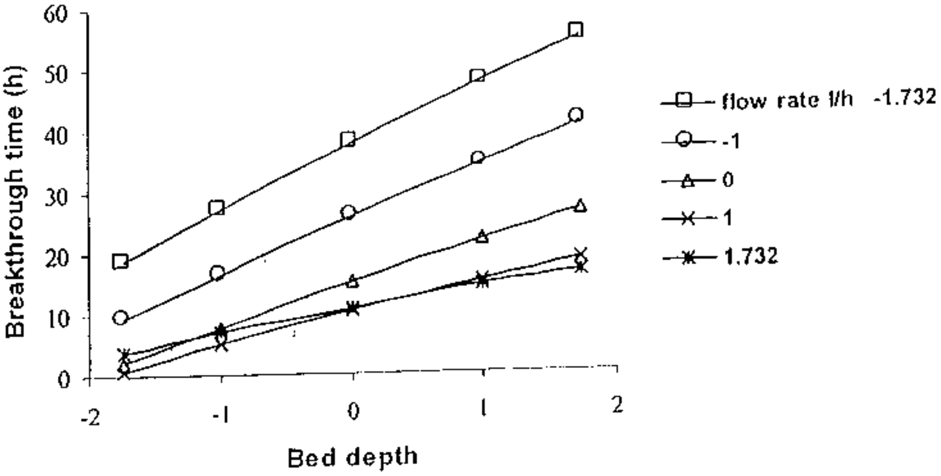


Fig (4.26) Effect of bed depth and flow rate on breakthrough time

4.6.2 Effect of Studied Variables on Adsorption Capacity

Once more the effect of three variables on adsorption capacity in Al-Zawraa wastewater experimental investigation have been emphasized. To demonstrate the effect of each variable on adsorption capacity clearly and individually, each variable was studied separately from the other variables as they kept at constant values (i.e. optimum conditions). As shown in Figs (4.27) to (4.29) the effects of studied variables (i.e., total dissolved solids concentration, flow rate and bed depth) on the adsorption capacity were investigated. These figures indicate that adsorption capacity of the ion exchanger is increased with increase in TDS concentration, and bed depth and decreased with higher flow rates. The reasons for these characteristics are attributed as follows:

Fig (4.27) shows variation of adsorption capacity with TDS concentration since adsorption capacity depends on a number of process variables including ionic concentration that leads to greater adsorption capacity as a result of higher mass transfer rate.

Fig (4.28) demonstrates the effect of flow rate on adsorption capacity that shows lower adsorption capacity with higher flow rates. This was directly contributed to higher mass transfer rates through the film and consequently the leakage curve is spread out over deeper reaction zone that might shorten exhaustion time and lower adsorption capacity.

In Fig (4.29), the adsorption capacity was increased with longer bed height. It was simply observed that longer column means greater adsorption capacity, which is simply attributed to the deeper, is the reaction zone.

Interaction effects between the studied variables have noticed in Figs (4.30) to (4.32). The effect of flow rate on the adsorption capacity at different total dissolved solids concentrations was studied in Fig (4.30). In this figure the adsorption capacity decreases with increasing flow rate at the highest limit of TDS concentration. In contrary, at the lowest limit of TDS concentration, adsorption capacity increases with flow rate. This can be attributed at the lower concentration of copper ions faster uptake of Cu^{+2} ions might occur that resulted to higher adsorption capacity with flow rate increase.

In Fig (4.31) the interaction effect between TDS concentration and bed depth has shown slight increase in adsorption capacity. It shows that at low TDS concentration, lower adsorption capacity was noticed. This can be attributed to pronounce selectivity of Cu^{++} ions at higher concentration that resulted in higher adsorption capacity.

Figure (4.32) demonstrates the interaction effect between flow rate and bed depth. At higher values of flow rate, large variation in adsorption capacity with bed depth was noticed in comparison with the lower values. However, at low values of flow rate, higher adsorption capacity was observed in comparison with higher values. This observation had been already noticed and explained latter according to slightly diffuse wave front that result of each successive layer of resin at low flow rate being almost completely exhausted before leakage occurs. As flow rate increases, equilibrium is no longer reached and the exhaustion curve is spread out over deeper reaction zone which shows lower adsorption capacity.

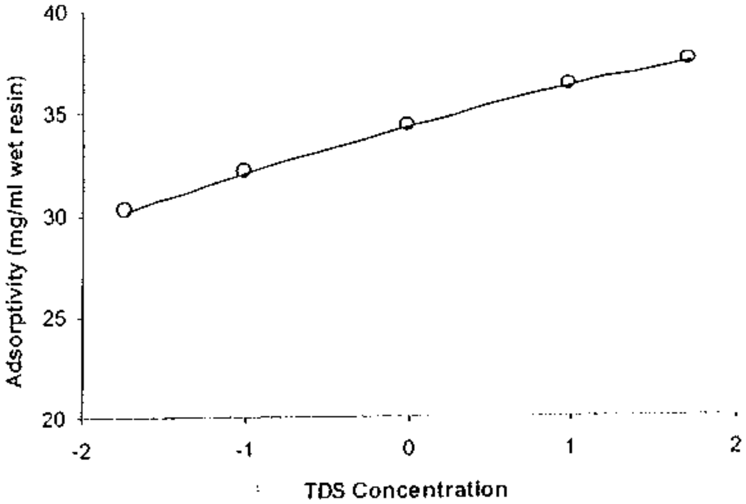


Fig (4.27) Effect of TDS concentration on adsorption capacity

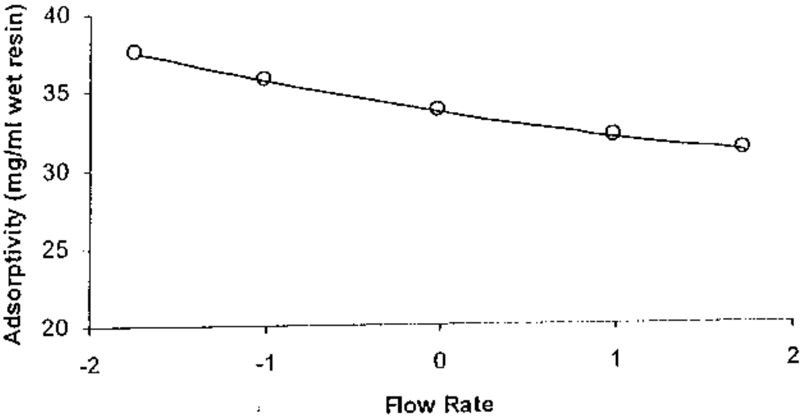


Fig (4.28) Effect of flow rate on adsorption capacity

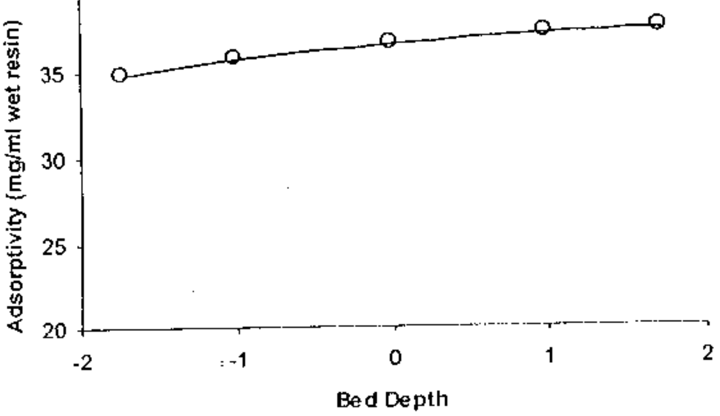


Fig (4.29) Effect of bed depth on adsorption capacity

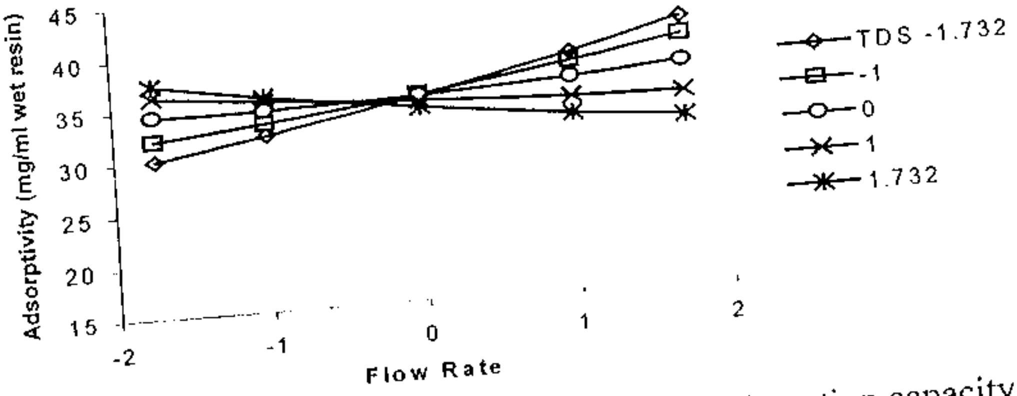


Fig (4.30) Effect of flow rate and TDS concentration on adsorption capacity

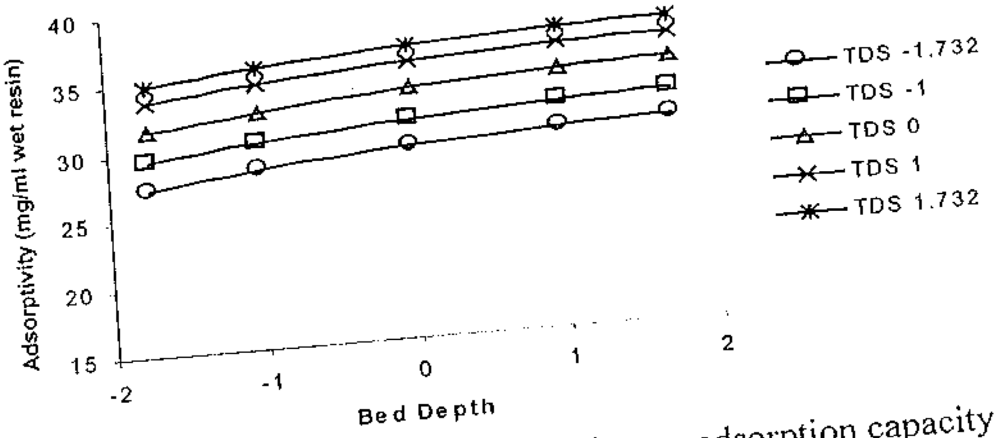


Fig (4.31) Effect of bed depth and TDS concentration on adsorption capacity

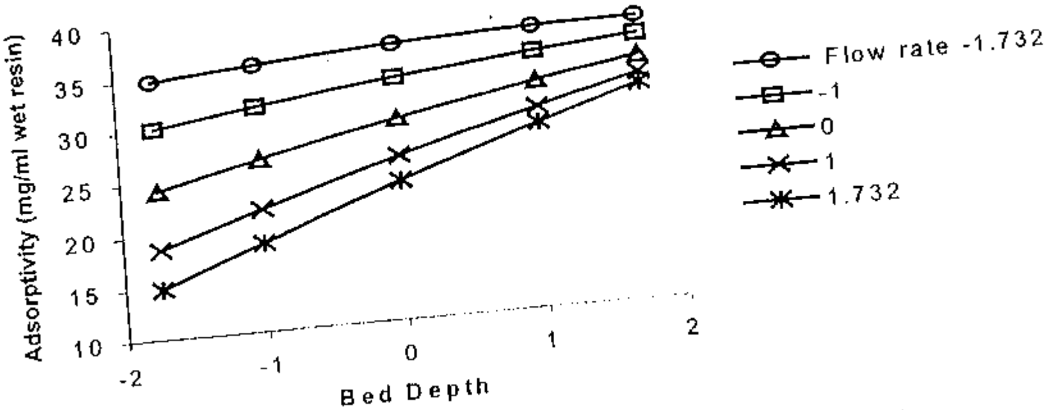


Fig (4.32) Effect of bed depth and flow rate on adsorption capacity

Chapter

5

Chapter Five

Conclusions and Recommendations

Chapter Five

Conclusions and Recommendation For Future Work

5.1 Conclusions

The following conclusions can be pointed out from this study:

1. Study the characteristic performance of production of demineralized water via ion exchanger, two mathematical correlations representing two objective functions i.e., the adsorption capacity and breakthrough time, had been correlated with four variables that representing the most affected parameters (i.e., copper concentration, TDS, feed rate and bed depth) had been developed and adequately describe the behavior of the process throughout the studied ranges of studied variables.

2. Study the individuality of wastewater from Al-Zawrra Company by ion exchangers three variables (i.e., TDS, feed rate and bed depth) had been correlated with two objective functions, the adsorption capacity and breakthrough time that adequately describe the characteristic performance of the ion exchanger.

3. It was shown that the studied four variables of the first set of experiments affects the adsorption capacity and breakthrough time in following sequence: $X_4 > X_3 > X_2 > X_1$ and $X_3 > X_4 > X_2 > X_1$ respectively.

4. It was shown that the studied three variables of the second set of experiments (i.e., Al-Zawraa wastewater treatment) affects the adsorption capacity and breakthrough time in following sequence: $X'_3 > X'_2 > X'_1$ and $X'_2 > X'_3 > X'_1$ respectively.

5. It seems that increasing the total dissolved concentration leads to lesser breakthrough time or breakthrough time is inversely related to concentration i.e., the breakthrough time was decreased with increasing the inlet concentration of total dissolved solids of Al-Zawraa Company wastewater.

6. Pronounced effect of bed depth on the breakthrough time and adsorption capacity was found.

7. The effect of flow rate on breakthrough time and adsorption capacity indicates that higher flow rate leads to lesser breakthrough time which means higher amounts of total dissolved solids adsorbed at lower flow rate for a given time besides it may contribute to lower residence time inside the column.

8. It was ascertained that various criteria influencing the general performance of ion exchange resin as the strong cation resin operating in the H^+ form will preferentially exchange ions with different selectivity that resulted in earlier breakthrough time with increase in concentration of copper ions due to its higher selectivity in comparison to Na^+ ions.

9. Higher concentration of total dissolved solids leads to higher concentration of ions in solution that resulted to higher mass transfer rate through the film until it exceeds the diffusion rate through the resin beads and consequently the system is exhibited particle controlled kinetics that means deeper reaction zone (a condition that leads to shorter exhaustion time and lower operating capacity).

10. With slow flow rate, it seems that the wavefront is only slightly diffuse as a result of each successive layer of resin being almost

completely exhausted before leakage occurs and resulted to a smaller operating capacity and earlier breakthrough point till no tendency to decrease at higher flow rates where the process exhibits particle controlled kinetics where no influences might contributed from further increase in flow rate.

11. Optimum conditions at the highest adsorption capacity and breakthrough time as determined for the first set of experimental work, Eq (4.1) and Eq (4.2) are found equal to 21 ppm copper concentration, 250 ppm TDS concentration, 5.097 l/h feed rate, and 70 cm bed depth and 5 ppm copper concentration, 250 ppm TDS concentration, 0.38 l/h feed rate, and 70 cm bed depth, respectively.

12. Optimum conditions at the highest adsorption capacity and breakthrough time as determined for the second set of experimental work, Eq (4.3) and Eq (4.4) (i.e., Al-Zawraa wastewater treatment) are found equal to 1193 ppm TDS concentration, 0.712 l/h feed rate, and 60.64 cm bed depth and 317 ppm TDS concentration, 0.712 l/h feed rate, and 60.64 cm bed depth, respectively.

5.2 Recommendations for Further Study

1. In the light of the results from this study, an extended study might arise to deal with the production of demineralize water using the mixed bed ion exchanger.

2. Before taking advantage of the results of this study to be implemented in situ, and to be in good position, an extended study might investigate other pollutants with larger concentrations.

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الدرجة الثانية لربط كفاءة اداء المبادلات الايونية بدلالة القابلية الامدصاصية وزمن الاستنفاذ كلاً على حده بالمتغيرات الاربعة للمرحلة الاولى وبالمتغيرات الثلاثة للمرحلة الثانية.

تم قياس القابلية الامدصاصية وزمن الاستنفاذ عملياً باستخدام محلول يحتوي على ايونات النحاس بتركيز يتراوح من 5 الى 25 جزء بالمليون وتركيز الاملاح الذائبة من 250-1250 جزء بالمليون ومعدل جريان المغذي من 0.38 الى 5.34 لتر بالساعة وعمق الطبقة من 5 الى 70 سم. كما تم قياس القابلية الامدصاصية وزمن الاستنفاذ عملياً باستخدام مياه الفضلات المطروحة من شركة الزوراء العامة بالظروف التالية: تركيز الاملاح الذائبة من 317 الى 1193 جزء بالمليون، معدل جريان المغذي من 0.712 الى 4.98 لتر بالساعة وعمق طبقة مبادل الايونات الموجبة من 9.4 الى 60.6 سم وطبقة مبادل الايونات السالبة من 14.3 الى 91.0 سم.

افترضت الدراسة اربعة معادلات متعددة الحدود من الدرجة الثانية واوجدت حدودها بتطبيق طريقة تحليل الارتداد اللاخطية على النتائج العملية لربط المتغيرات الاربعة للدراسة الاولى والمتغيرات الثلاث للدراسة الثانية بالقابلية الامدصاصية وزمن الاستنفاذ.

اشارت النتائج العملية المستحصل عليها من الدراسة كفاءة استخدام منظومة التبادل الايوني المتكونة من مبادلين الاول مبادل الايونات الموجبة الحامضي القوي بصيغة (H^+) والثاني مبادل الايونات السالبة القاعدي الضعيف بصيغة (OH^-) للتخلص من كافة العناصر الثقيلة السامة والايونات السالبة المرافقة لها وكافة الايونات الموجبة والسالبة للاملاح الذائبة في الماء.

إنتاج الماء اللاايوني
من مياه فضلات معمل إنتاج البطاقات الإلكترونية
بواسطة المبادلات الايونية

إعداد
المهندس مظفر يعقوب حسين

تحت إشراف
الدكتور المهندس ناهض وديع ميخائيل
الدكتور مثيل ضايف السبتي

الخلاصة

يهدف البحث الى دراسة العوامل المؤثرة على اداء المبادلات الايونية الموجبة والسالبة لمعالجة المياه المطروحة من مصانع انتاج الدوائر الالكترونية المطبوعة واسترداد الماء على شكل ماء لاايوني عالي النقاوة. يضاف الى ذلك دراسة معالجة محلول تنشيط مبادل الايونات الموجبة بتطبيق تقنية ترسيب ايونات النحاس. تم دراسة استرداد ومعالجة مياه الفضلات من خلال قياس خاصيتين للمبادلات الايونية وهي القابلية الامدصاصية وزمن الاستنفاذ. لدراسة اداء المبادلات الايونية الموجبة والسالبة تم تعريض مياه الفضلات من مصانع البطاقات الالكترونية الى نوعين من المبادلات الايونية (مبادل الايونات الموجبة القوي الحامض ومبادل الايونات السالبة الضعيفة القاعدة). قسمت الدراسة الى دراستين: الاولى تقوم بدراسة اداء المبادلات الايونية لانتاج ماء لاايوني من نماذج مياه محضرة مختبرياً وتأثير اربعة متغيرات مختلفة وهي تركيز النحاس، تركيز الاملاح الذائبة الكلية، معدل جريان المغذي وعمق الطبقة. أما الدراسة الثانية فشملت دراسة استجابة المبادلات الايونية الموجبة والسالبة بانتاج ماء لاايوني من مياه الفضلات لمصنع البطاقات الالكترونية في شركة الزوراء العامة مع دراسة تأثير ثلاث متغيرات وهي تركيز الاملاح الذائبة الكلية ومعدل الجريان وعمق الطبقة. استخدمت طريقة بوكس ويلسن في تصميم التجارب العملية وافترضت علاقات رياضية متعددة الحدود من