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The Characteristic Performance of Fluidized bed Ion Exchange

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Abstract

The aim of this work is to study the characterization of ion exchange in important characteristics (i.e., breakthrough time and exchange capacity) by investigation the performance of cation exchange column subjected to makeup solution in term of three different parameter (i.e., bed depth 5-45cm, NaCl salt concentration100-2000 ppm and feed flow rate 9.5-26.5l/h).

Two second order polynomial model are proposed with Box Wilson composite rotatable design. The experimental result had shown an efficient performance of using ion exchange column with strong acid cation exchange resin in fluidized mode operation which given maximum breakthrough time and higher exchange capacity means the rate of exchange is very rapid to completed the removing of all undesired ions at a condition of low bed depth, high NaCl concentration and high feed flow rate.

Keywords: Fluidization, Ion Exchange

Introduction

Ion exchange is a process in which mobile ion from an external solution are exchanged from ions that are electro statically bound to the functional group contained within a solution matrix. Ion exchange is widely used technique for separation of ion from different solution where the process is required for the treatment effluent of the cases that ion to be removed exists at very low concentration together with other ions.(International Atomic Energy Agency 2002)

Fluidized bed which consist of up flow column containing resin represent the perfect way to operate column without moving the resin out the column where thewater flows up words through the resin bed, velocity must be adjusted to obtain the level of bed expansion required to allow unrestricted free passage of concentrated solution. Resin fills most of the volume contained between the upper and lower plates that serve to strain and evenly distribute water and make the resin uncompressed and free to float through the bed during the exchange phase. (Dyer and Hudson 1997) The chief advantage of the fluidization is that the solid is vigorously mixed by the fluid passing through the bed and mixing of solid ensures where practically no temperature gradients in the bed. Furthermore, the particle is capable of giving good quality fluidization that gives high mass transfer for that reason, it is important to study the characteristics of fluidizing mode operation to give better performance. (Applebaum 1968)

Sreat and Naden (1987) indicated the operation requires that the solution residence time should be as short as possible and flow/unit area as high as possible and the solution flow rate must take the resin fluidization into account.

The sample application of such column is the conversion of salt of NaCl into an equivalent amount of acid by exchange with the hydrogen form of a cation exchange resin.

$NaCl_{aq}$ +H-R → Na-R + HCl_{aq} ………(1)

The strong acid resin is regenerated with hydrochloric acid and rinsed with demineralized water.

Column performance in these processes is commonly described in term of effluent concentration histories or break through curves which display the concentration of ion versus time. (Rem CO. 2000)

A solution of Na^+ ions (as NaCl) is passed through a column containing AMBERJET 1200H cation resin (for exchanging positively charged ions) in the form of spherical beads. This resin can adsorb Na^+ ions from solution and return in exchange the same number of H^+ ions, so that the solution maintains electrical neutrality.

As the solution passes through the column, it becomes more dilute in Na^+ and more concentrated in H^+. Eventually the resin becomes saturated with Na^+ ions and further exchange ceases, the ion exchange process is reversible, so that the resin can be regenerated by allowing it to come in contact with a strong acid (HCl).

De Silva (1994) found that separated bed system features 98-99% removal with effluent sodium 0.5-10 ppm, conductivity 2.5-50 µmhos.

Sreat and Naden (1987) indicated the operation requires that the solution residence time should be as short as possible and flow/unit area as high as possible and the solution flow rate must take the resin fluidization into account.

Macub (2002) suggests that the flow is usually continuous but pulsing can also increase the rate of mass transfer. A number of systems have used the pulsing technology which additionally helped to fluidize the resin.

Streat and Naden (1987) conducted an important work on fluidized bed operation they also studied the effect of feed flow rate which is usually chosen to give a bed expansion of less than 200% .

because higher flow rate will lead to resin carry over.

Willkes (2002) studied the characteristics of fixed and fluidized bed ion exchange with strong acid cation exchange column and proposed a model with pH effect to study exchange capacity.

Experimental Design

The purpose of an experimental design is to find the useful relationship between the controllable and observed response with minimum number of experiments where the analysis of experiments will give description for the system. (Montgomry1976)

Box-Wilson composite rotatable design is one of statistically experimental design which is a series of experiment that have been developed for second order response surfaces.

General form of second model polynomial is illustrated by equation:

$$Y = A_o + \sum_{i=1}^{ki} A_{i} X_{i} + \sum_{i=1}^{ki}\sum_{j=1}^{ki} B_{ij} X_{ij} + \ldots \text{for } i > j$$

ki = number of variables in the system

Y = objective function

The preliminary step is to setup the relationship between coded level and corresponding real process variable which is required in the determination of experimental by the following equation:

No. of Exp. $=2^{ki}+2ki+1$

ki= variable =3

No of exp. $=2^3+2*3+1=15$

For purposes of estimation of experimental error, the center point is usually repeated three times during the experiment. (Jeffwn and Hamada 2000)

Experiments are carried out due to the experience of the experiments and the most suitable range of the studied condition within a reasonable range to determine the fluidizing velocity in order to given the operation rang of flow rates (9.5 – 26.5 l/h), bed depth of fluidizing without moving out the column (5 - 45 cm) and the higher range of NaCl concentration (100 - 2000 ppm).

Total No. of exp. =15+3=18

Variable:

X_1 = Bed depth of cation resin (5- 45 cm)

X_2 = Concentration of NaCl solution (100 - 2000 ppm)

X_3 = Flow rate (9.5 – 26.5 l/h)

Experimental Work

A schematic diagram of experimental apparatus employed through this work is shown in figure (1). It simply consists of the following parts:

*The ion exchange column is the principal part used in the work, it consist of (Q.V.F) glass pipe of i.d (2.45 cm) and height (1.2 m), the column was filled with strong acid cation resin of (AMBERJET 1200 H). The resin was supported inside the column by a glass dish welded to the walls of the column on the bottom section; the glass support is incorporated with holes of 0.1μmm inside diameter placed in a conical shape Teflon

The column is incorporated with 11(Q.V.F) valves of shut-off type, these valves are located in different positions in order to coordinate the flow of NaCl solution, regenerate solution (HCl) and the leaving of deionized water during the different steps of process; the valves are shown in a schematic diagram in table (1).

Four plastic vessels are assigned. The first is for feed solution (NaCl), the second is for regenerate solution (HCl) acid, the third is for deionized water and the fourth is for the resultant solution from the column.

* A dosing pump with a fine adjustment scale of (0-1000) are used for flow control with a capacity rate of (0-30 l/h) and pressure capacity of (10) bar.

*A digital pH meter was used in the inlet bed and exit for measuring the pH values as function of time

*NaCl, HCl solutions and deionized water were measured separately using a calibrated rotameter model1100 type GEC-Elliott.

*Two stainless steel oil type gauges were used in the top and in the bottom of the column to determine the pressure drop across the column with scale of (0-2) bar with NKS type.

Experiment procedure

Exhaustion

Eighteen experiments shown in tables (2) were carried out with different (bed depth, NaCl concentration and flow rate). All these runs followed the same procedure where NaCl solution is pumped via pump (P) from tank (1) into column (C) upward in fluidized bed operation until break point is happened when no variation of hardness.

Backwash

The cation exchanger in column C was washed upwardly with a flow rate of 10 bed volumes for 10 to15 min using deionized water from tank 3 in order to remove any particulate material, clear the bed of any air pocket or bubble and reclassify the resin particle. During the upward flow backwash, the resin bed should be expanded in volume from 50% to 80% of its initial value.

Regeneration

Regeneration of the cation exchange is carried out by 4% HCl solution. The acid solution is pumped from tank T_2 through pump (P) into column C downward with flow rate of 4 bed volumes. The regeneration normally took 75 min.

Rinse

Through this step, downward deionized water with 4 bed volumes is pumped from tank T_3 through pump P to cation exchange column C. This step took 30 minutes which it called slow rinse.

Then this step is accomplished with downward deionizing water of 10 bed volumes from tank T_3 to cation exchange column C. This step took 90 minutes which it called quick rinse.

Results and Discussion

Effect Bed Depth on Breakthrough Time

The breakthrough time is plotted at figure (2) at different bed depths with a flow rate of (9.5 l/h) and NaCl concentration of (100ppm), Figure (2) shows that the breakthrough time increases with the increase of bed depth because longer bed depthgives longer mass transfer zone, which mean additional opportunity will be available for salt ions to be exchanged with the H^+ ion in the resin.

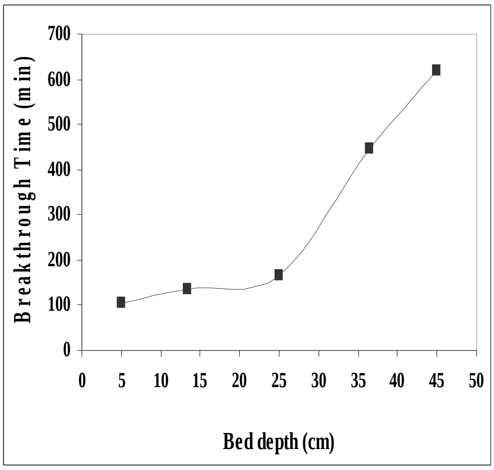


Fig.(2) Effect of bed depth on breakthrough time

Effect of NaCl Concentration on Breakthrough Time

Figure (3) show that the breakthrough time is plotted at different salt concentration with bed depth (0.45m) and a flow rate (9.5l/h). The figure shows that the breakthrough time decreases with the increase of NaCl salt concentration, with higher concentration the concentration of the ions in the solution increases which raises the mass transfer rate through the film until it exceeds the diffusion rate through the resin beads. Then the diffusion rate becomes the controlling factor and the system is said to exhibit particle controlled kinetics, which is a slower process.

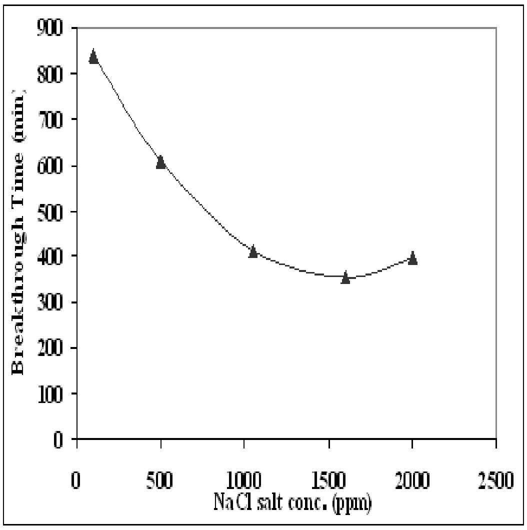


Fig. (3) Effect of NaCl concentration on breakthrough time

Effect of Flow Rate on Breakthrough Time

The breakthrough time is plotted at figure (4) at different flow rates at bed depth of (0.45m) and NaCl salt concentration (100ppm), it seem that the break through time decreases with the increase of flow rate because at lower flow rate there will be sufficient contact time for Na^+ ions to exchange with H^+ ions in the resin particles thus the equilibrium is established soon. As the flow rate increases equilibrium is no longer reached and the mass transfer through the film rises until it exceeds the diffusion rate through the resin bed.

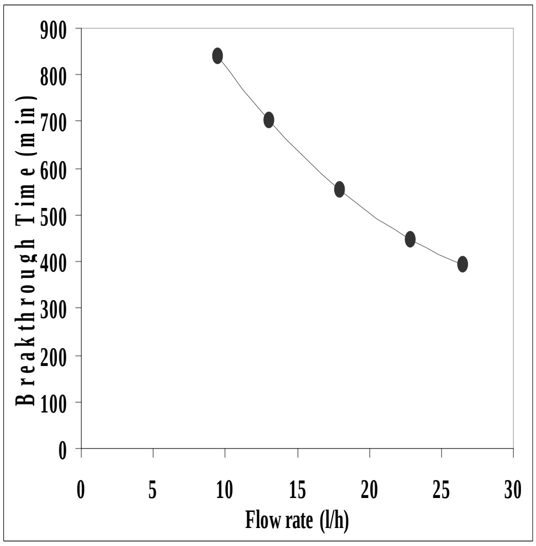


Fig. (4) Effect of Flow Rate on breakthrough time

Effect of Bed Depth on Exchange Capacity

The exchange capacity is plotted at figure (5) at different bed depths with a flow rate of (26.5l/h) and NaCl concentration (2000ppm). It shows that the exchange capacity decreases with the increase of bed depth this can be explained when bed depth decreases it becomes lighter in weight and easier to expand and the particles are more vigorously agitated by the fluid passing through the bed and the mixing of the particle is efficient and more directly in contact with the fluid which leads to more mass transfer between the resin and Na^+ ion thus increases the mass transfer zone and increases exchange capacity.

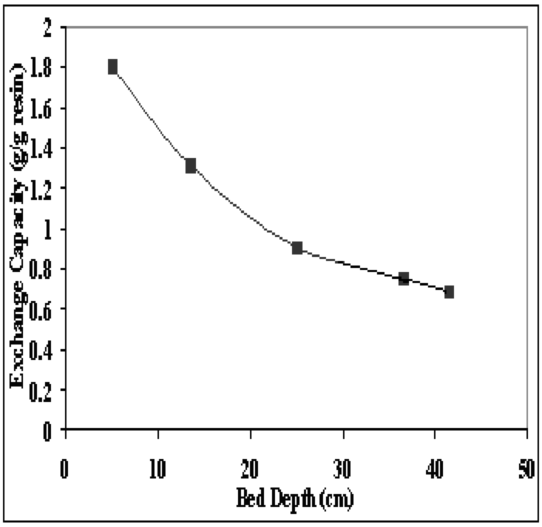


Fig. (5) Effect of Bed depth on exchange Capacity

Effect of NaCl Salt Concentration on Exchange Capacity

The exchange capacity is plotted at figure (6) at different salt concentrations at bed depth of (0.05m) and flow rate (26.5l/h) this figure shows that the exchange capacity increase with the increase of NaCl salt concentration It was simple to observe that higher NaCl salt concentration leads to greater operation capacity as a result of greater reaction zone concentration in solution around the resin particle. At higher concentrations of NaCl, the uptake of Na^{+} ions might be faster which causes to lesser breakthrough time and leads to greater adsorption capacity as a result of high mass transfer.

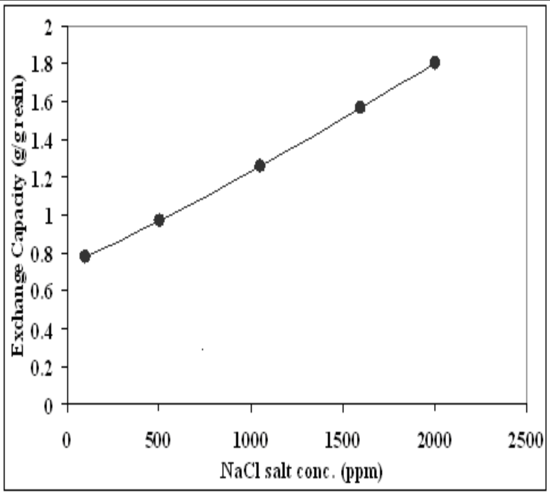


Fig. (6) Effect of NaCl concentration on Exchange Capacity

Effect of Flow Rate on Exchange Capacity

The exchange capacities is plotted at figure (7) at different flow rates at bed depth (0.05m) and NaCl salt concentration (2000ppm) this figure shows that the exchange capacity increases slightly with the increase of flow rate this relation can be explained that as flow rate increases the mixing inside the column increases which prevents the collocation of resin particles in separated package group, which if a decreases happens in mass transfer area of the resin will lead to decrease total mass transfer and decrease exchange capacity.

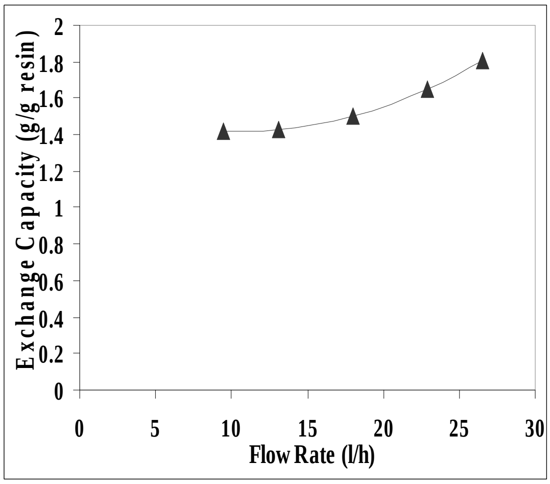


Fig. (7) Effect Flow Rate on Exchange capacity

Conclusion

1. Increasing the bed depth leads to long breakthrough time.
2. The break through time decreases with the increase of NaCl salt concentration.
3. The effect of influent flow rate on breakthrough time indicates that higher flow rate leads to lesser breakthrough time.
4. Increasing bed depth leads to decreasing the exchange capacity on fluidized bed ion exchange, because in high bed depth the resin collocates and works as package group work separately on each other which decreases the mass transfer and lower exchange capacity.
5. As the concentration increases the exchange capacity increases because high concentration means high concentration gradient in the hydraulic film around the resin particle
6. The exchange capacity increases as flow rate increases because high flow rate means more bed expansion more fluidizing effect that increases the surface area of the mass transfer of resin particle and the mass transfer of the whole fluidized resin bed leads to increase exchange capacity.

Reference

1. International Atomic Energy Agency (Application of Ion Exchange Processes for the Treatment of Radioactive Waste and Management of Spent Ion Exchange) “Technical Report Ser No. 408” 2002 Vienna

2. Dayer and Hadson, (ion Exchange and Fluidized bed), www.ion exchange.com 1997

3. Applebaum (Demineralization by Ion Exchange), United kingdom third Edition published USA 1988

4. Streat and Naden, (Ion Exchange and Sorption Processes in Hydrometallurgy Critical), Report on Applied Chemistry, Vol.19, Society of Chemical Industry, Jon-Wielly&Son, 1987

5. REMCO Engineering (Water System and Control), www.REM.Eng.com

6. De Silva (Ion Exchange Deal), Chemical Engineering, July 1994

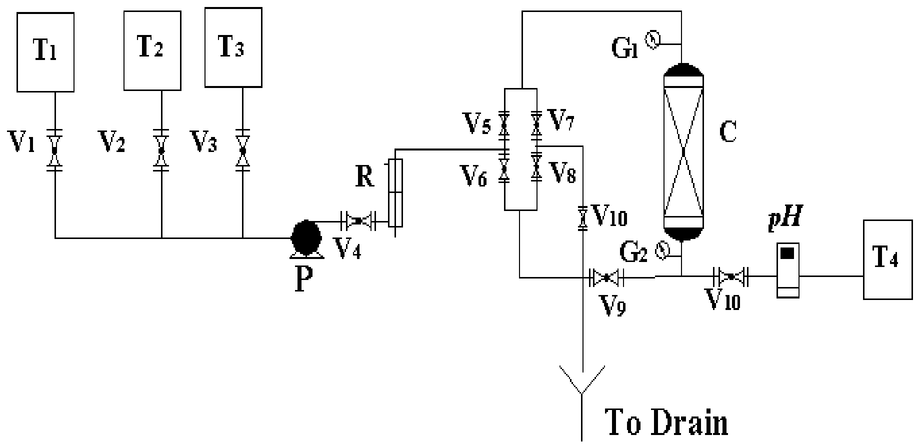
7. Flint box co., www.Flint box.-technology Introduction continuous bed ion exchange system 2004

8. Macab, (Unit Operation of Chemical Engineering), 7th Edition,McGraw-Hill, 2005

9. J.O. Wilkes and Pablo LaValle (Fixed and Fluidized Bed Ion Exchange), University of Michigan ChE 360 Laboratory, 12-2002, www.Michigan

10. D.C. Montgomry, (Design and Analysis of Industrial Experiments), John-Wily and Sons, New York, 1976

11. C.F. Jeffwn and Mechael Hamada, (Experiments Planning Analysis), John-Wily and Sons, New York, 2000



ITEM	DESECRIPTION
T_1	NaCl salt tank
T_2	HCl tank (4%)
T_3	Deionized water tank
T_4	Sampling result tank
P	Pump
R	Rotameter
pH	pH meter
C	Cation exchange column
V	Valve
G	Pressure gage

Figure (1) schematic diagram of system of experimental work

Table (1) Configuration of valves through service life of ion exchange

Name of Step	Valves No.										
	V_1	V_2	V_3	V_4	V_5	V_6	V_7	V_8	V_9	V_{10}	V_{11}
Fluidized bed Exhaustion	O*	C**	C	O	C	O	C	C	O	C	O
Back wash	C	C	O	O	C	O	O	C	O	O	C
Regeneration	C	O	C	O	O	C	C	O	O	O	C
Rains	C	C	O	O	O	C	C	O	O	C	O

Table (2) Coded and Real Variables of Experiments to be conducted according to Box- Wilson method

Run No.	Coded Variable			Real Variable			Cation Volume (ml)
	X1	X2	X3	Bed depth (cm)	Conc. of NaCl (ppm)	Flow rate (l/h)	
1	+1	+1	+1	36.5	1598.5	22.9	172
2	-1	+1	+1	13.4	1598.5	22.9	63.17
3	+1	-1	+1	36.5	501.5	22.9	172
4	-1	-1	+1	13.4	501.5	22.9	63.17
5	+1	+1	-1	36.5	1598.5	13.09	172
6	-1	+1	-1	13.4	1598.5	13.09	63.17
7	+1	-1	-1	36.5	501.5	13.09	172
8	-1	-1	-1	13.4	501.5	13.09	63.17
9	1.732	0	0	45	1050	18	212.15
10	0	1.732	0	25	2000	18	117.86
	0	0	1.732	25	1050	26.5	117.86
12	-1.732	0	0	5	1050	18	23.57
13	0	-1.732	0	25	100	18	117.86
14	0	0	-1.732	25	1050	9.5	117.86
15	0	0	0	25	1050	18	117.86
16	0	0	0	25	1050	18	117.86
17	0	0	0	25	1050	18	117.86
18	0	0	0	25	1050	18	117.86

دراسة محددات اداء العمود الايوني ذو الطبقة المسيلة

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الخلاصة

يهدف البحث المقدم الى دراسة العوامل المؤثرة في اداء المبادلات الايونية من خلال قياس خاصيتين مهمتين هما زمن الاستنفاذ وقابلية التبادل عن طريق بحث اداء عمود للمبادلات الايونية الموجبة معرض لمحاليل مياه محضرة مختبريا من خلال ثلاثة متغيرات مختلفة (ارتفاع الطبقة ٥-٤٥ سم ، تركيز محلول ملحي من كلوريد الصوديوم ١٠٠-٢٠٠٠ جزء بالمليون ومعدل جريان المغذي ٩,٥- ٢٦,٥ لتر/ساعة).

ُافترضت معادلتان متعددة الحدود من الدرجة الثانية واوجدت حدودها بطريقة بوكس ويلسن للتحليل الارتدادي اللاخطي .وقد اظهرت النتائج العملية كفاءة استخدام عمود المبادل الايوني الموجب الحامضي القوي بطريقة الطبقة المسالة والذي يعطي اعلى زمن للاستنفاذ واعلى قابلية للتبادل والذي يعني ان معدل التبادل سريع جدا للقيام باكمال ازالة جميع الايونات غير المرغوبة تحت ظروف تشغيلية اقل ارتفاع للطبقة،اعلى تركيز لكلوريد الصوديوم واعلى معدل جريان للمغذي.