

Full Length Research Paper

New technological root for regenerating demineralized water plants for safe environment

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The experiments in this work were subjected to lab-scale ion exchangers unit, consists of two kinds of ion exchangers (strong acid cation exchanger and weak base anion exchangers). The experimental investigation was directed at studying a new method for regenerating the strongly acidic cation exchanger in the demineralized water unit using one concentration of sulfuric acid (5%) directly without any precipitation problems. Experiments show that using sulfuric acid in the regeneration is equally efficient as that of hydrochloric acid solution in the production of dematerialized water (110 bed volume). The first objective of this study was to use sulfuric acid solution instead of hydrochloric acid solution because it is cheaper and non corrosive. The other benefits could be gained from the effluent (sodium hydroxide solution) which comes out of the anion exchanger columns instead of disposing them.

Key words: Cation exchanger, anion exchanger, backwash, regeneration, rinsing.

INTRODUCTION

Ion exchange is a chemical treatment process used to remove unwanted ionic species from water and wastewater. It is basically a simple process based on reversible interchange of ions between liquid and solid (that is, the resin) with no permanent changes in the structure of the solid (ABB, 1999). 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 (Boloto and Pawloeski, 1987). Water treatment, however, is the most important application of ion exchange. The main areas of interest are water demineralization or de-ionization (the product of high purity water). Deionization removes all types of ions from the water, leaving only non-ionic materials in the solution (Remco, 2000).

Ion exchangers are insoluble solid particles which are either naturally occurring inorganic zeolite or synthetically produced organic resins. Most of the polymer bases used for ion exchange resins in beads form are the most widely utilized materials in many fields of science and industry (Alberti, 1995; luca, 2000). The main types of synthetic ion exchangers were obtained by polymer-analogous reactions performed on porous S-DVB copolymers (Dragan, 1987; 2004). These resins have been classified based on the charge in the exchangeable

counter ion (cation exchanger or anion exchanger) and the ionic strength of the bound ion (strong exchanger or weak exchanger) (Aldrich, 2002).

This work used one concentration of H₂SO₄ solution (5%) directly without precipitation problems but with new root for solutions flow; this reduced the regeneration time by 40% compared with the gradual increase of H₂SO₄ concentration.

MATERIALS AND EQUIPMENTS

The materials used in this study include variety of chemicals such as H₂SO₄ (96%), NaOH solution (30%), HCl solution (30%), Strong acid cation exchangers (Duolite C20, Amberlite IR-120, Lewatit S-100), Strong base anion exchangers (Duolite A-101, Amberlite IRA-410, Lewatit MP-600) and weak base anion exchangers (Duolite A-368, Amberlite IRA-93, Lewatit MP-62).

Apparatus

The experimental rig consisted mainly of three deionized columns (that is, the cation exchanger and two anion exchangers). These columns were incorporated with dosing pumps. A schematic diagram of the apparatus is shown in Figure 1. Flame Atomic Absorption Spectrophotometer, Type Shimadzu AA – 680 G. Japan; pH meter, Type Titro-Processor 686 Metrohm Swiss made;

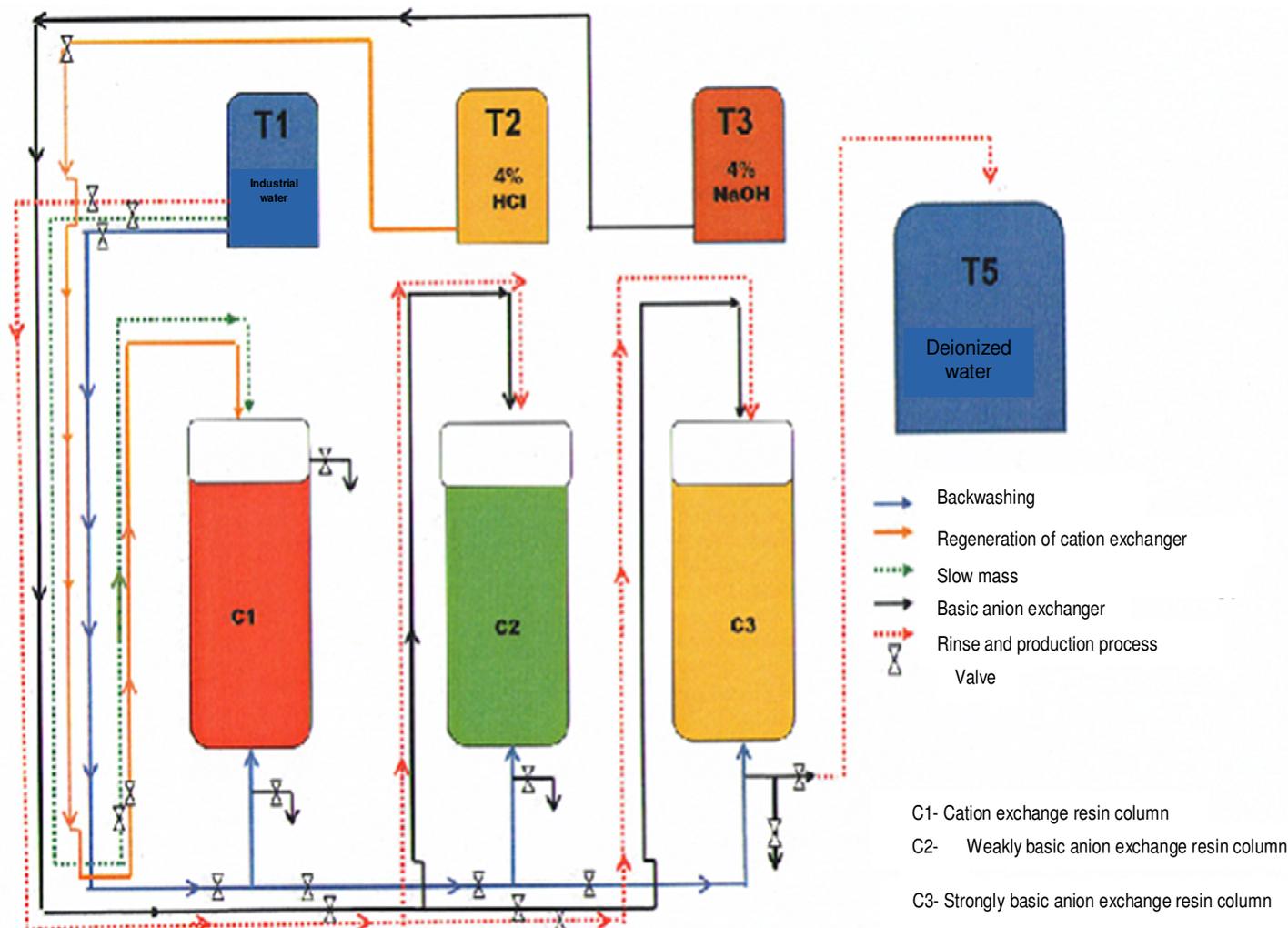


Figure 1. A simple diagram for the unit used for conducting the experiments where the dilute HCl solution (4%) was used as regenerant for the cation exchanger.

Conductivity meter, Type WTW LF91 Germany, ranged between 0 to 9000 $\mu\text{s}/\text{cm}$; Turbidity meter, Type HACH Company Model 43900 was used to measure the concentration of sulfate ions.

EXPERIMENTAL PART

Figure (1) expresses a simple diagram for the unit that was used for conducting the experiments where the dilute HCl solution (4%) was used as regenerant for the cation exchanger, while Figure 2 expresses a diagram where the dilute H_2SO_4 solution (5%) was used for the regeneration. Tanks (1, 2, 3, 4 and 5) containing: industrial water, dilute HCl solution (4%), dilute NaOH solution (4%), dilute H_2SO_4 solution (5%) and deionized water respectively. Six experiments were executed in order to get the optimum operating conditions to produce more quantity of deionized water of the best quality.

General procedure

Conditioning of the ion exchangers

Columns C_1 , C_2 and C_3 were packed with two-third of their volumes

by strongly acidic cation exchanger, weakly basic anion exchanger, and strongly basic anion exchanger respectively. The three columns were soaked with deionized water with the bed volume of two-third of the column volume (that is, exchanger packed without any bubbles exist between its particles).

Back washing

The cation exchanger in column C_1 was washed in an upwards direction (counter-current) with the flow rate 4-bed volume/ h for 10 to 15 min using industrial water from tank (T_1). Subsequently, the ion exchangers in the columns C_2 and C_3 were backwashed with the same flow rate as that for C_1 (Table 1).

Regeneration

Regeneration of the cation exchanger with 4% HCl: Regeneration of the cation exchanger was carried out by acid solution, HCl (4%). The acid solution was pumped from the tank T_2 downwards the column C_1 with the flow rate of 4-bed volume/ h

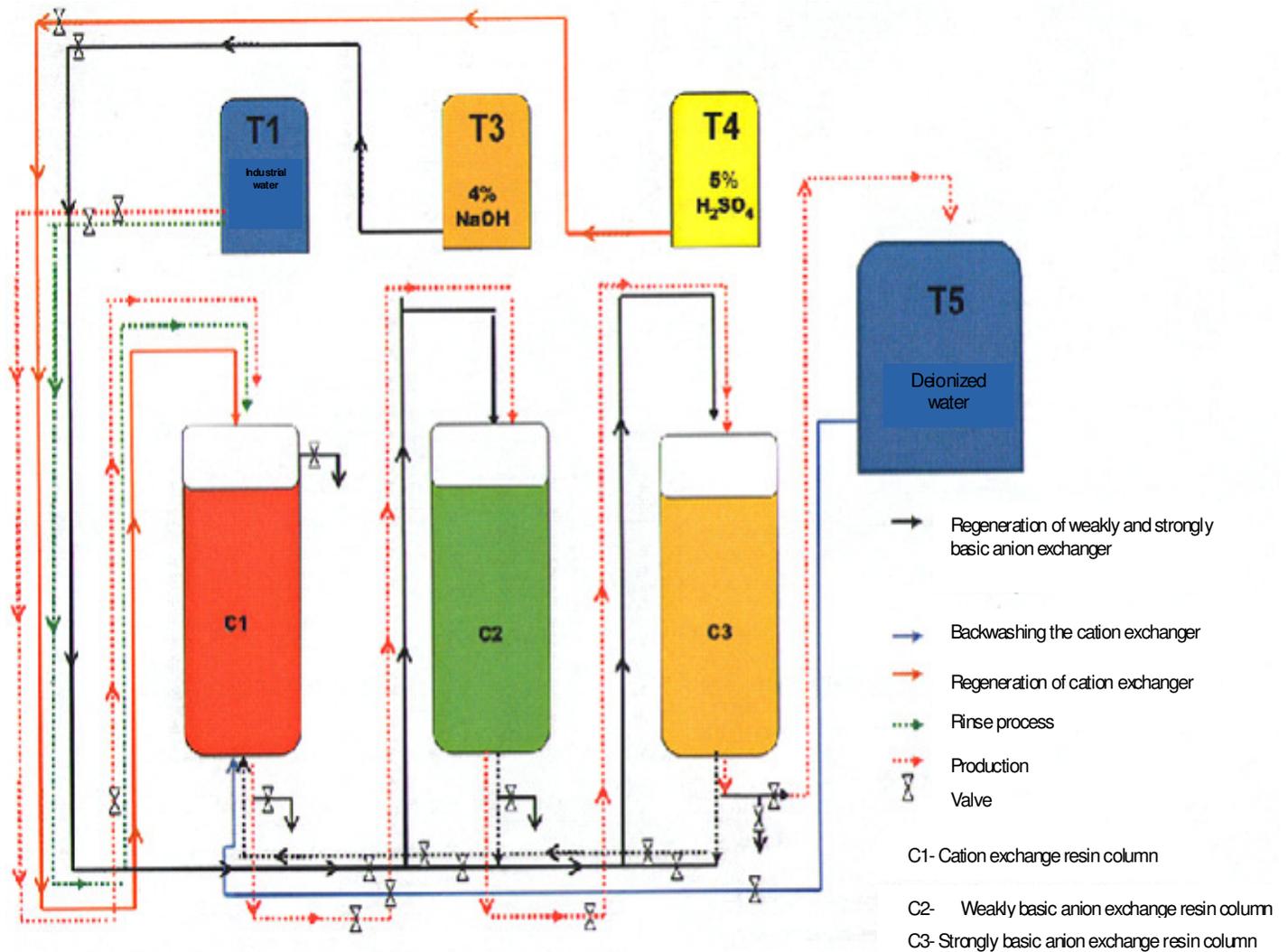


Figure 2. A diagram where dilute H₂SO₄ solution (5%) was used for the regeneration.

Table 1. The operating conditions for regenerating the cation exchanger using HCl and 5% H₂SO₄

Operation	Flow Rate		Solution		Time		Quantity	
	HCl (b.v./h)	H ₂ SO ₄ (b.v./h)	HCl	H ₂ SO ₄	HCl	H ₂ SO ₄	HCl (b.v.)	H ₂ SO ₄ (b.v.)
Back Wash	4	4	Ind. Water	Ind. Water	10	10	2/3	2/3
Ca ⁺⁺ Removal	-	8	-	NaOH (4%)	-	30	-	4
Rinsing	-	8	-	D. or soft water	-	15	-	2
Regeneration	4	4	HCl (4%)	H ₂ SO ₄ (5%)	30	45	2	3
Slow Rinse	4	4	Ind. Water	Ind. Water	15	15	1	1
Quick Rinse	4	8	Ind. Water	Ind. Water	30	20	4	8/3

(Table 1). The regeneration lasted 75 min.

Rinse process: Industrial water from tank T₁ was pumped downwards the column C₁ with the flow rate of 4-bed volume/ h for 15 min then the flow rate increased to 8- bed volume/ h for 30 min.

Regeneration of weakly and strongly basic anion exchanger: sodium hydroxide with concentration of 4% was pumped from the tank T₃ downwards the columns C₂ and C₃ with the flow rate of 4-bed volume/ h. The regeneration of anion exchangers lasted 45 min.

Table 2 .A comparison between the regeneration conditions by HCl and H₂SO₄

Exp. No.	Column C ₁	Column C ₂	Column C ₃	Regenerant	Average production	Efficiency (%)
1	C-20	A-368	A-101	HCl (4%)	115- b.v.	
2	C-20	A-368	A-101	H ₂ SO ₄ (5%)	110- b.v.	95.6
3	S-100	MP-62	MP-600	HCl (4%)	105- b.v.	
4	S-100	MP-62	MP-600	H ₂ SO ₄ (5%)	110- b.v.	104.7
5	IR-120	IRA-93	IRA-410	HCl (4%)	100- b.v.	
6	IR-120	IRA-93	IRA-410	H ₂ SO ₄ (5%)	100- b.v.	100

Rinse and production processes: Industrial water was pumped downwards through the column C₁ then to the top of the column C₂ and to the top of the column C₃ with the flow rate 4-bed volume/ h for 30 min and with the flow rate 8- bed volume/ h for 45 min. After the rinse process was finished, the flow rate was increased to 10-bed volume/ h and the effluent charged to the collection tank of deionized water T₅.

Regeneration of the cation exchanger by 5% H₂SO₄

(a) Regeneration of weakly and strongly basic anion exchanger: Sodium hydroxide with concentration of 4% was pumped from the tank T₃ downwards columns C₂ and C₃ with the flow rate of 4-bed volume/ h for 15 min and the effluents were disposed away. After that the effluents from columns C₂ and C₃ joined together and passed inside the column C₁ upwards with the flow rate 8-bed volume/ h for 30 min which was enough to regenerate the cation exchanger with sodium ions from the sodium hydroxide effluent (exhausted from hydroxide ions) and to expand the resin bed.

(b) Backwashing the cation exchanger: The deionized water from the tank (5) was used to wash the cation exchanger in column C₁ upwards with the flow rate 8-bed volume/ h for 15 min.

(c) Regenerating the cation exchanger: The H₂SO₄ solution (5%) from the tank (4) was passed downwards the column C₁ with the flow rate 4-bed volume/ h for 45 min.

(d) Rinsing the cation exchanger: Industrial water was pumped downwards the column C₁ with the flow rate of 4-bed volume/ h for 15 min then the flow rate increased to 8-bed volume/ h for 20 min.

(e) Production of demineralized water: Industrial water was pumped from the tank T₁ inside the columns C₁, C₂ and C₃, respectively downwards with the flow rate 10-bed volume/ h. The flow of the industrial water through the columns continued while the conductivity of the effluent stream was continuously monitored until breakthrough point was detected at the conductivity reading of less than (20 µs/cm) and the average pH (7.5 to 8) where the total productivity of deionized water was equal to 110 bed volumes.

RESULTS AND DISCUSSION

Table (1) represented the operating conditions for regenerating the strongly acidic cation exchanger using HCl (4%) and H₂SO₄ (5%) by the new method. This method enables usage of sodium ions in the effluents (normally disposed) which came out of the columns (C₂

and C₃) when regenerating the weak and strong basic anion exchangers. These effluents would flow upwards the column (C₁) where all the adsorbed cations (calcium, magnesium ...) on the cation exchanger would be exchanged for sodium ions in the effluent. In this stage the cation exchanger might be washed by deionized, decationized, or soft water. This enables precipitation of insoluble hydroxides to be avoided on the surface and in-between the ion exchanger layers when washed by industrial water. The precipitation would lead to pressure built up inside the column causing problems in the solutions flow decreasing the flow rate and the ion exchange capacity. However, when regenerating the cation exchanger by H₂SO₄ (5%), no precipitates occur, since the formed salt is water soluble. This was the aim of this work.

Table 2 represented all the experiments in order to select the best operating conditions and the model experiment. This table showed that the new root was better compared to the traditional one (HCl regeneration) and the productivity of deionized water was the same. The same table indicates in the model experiment, different kinds of ion exchangers were used in order to be applicable to most kinds of deionized water plants. Table 3 showed the concentration of cations and anions in the industrial and deionized water.

From the experiments one could notice that the first step in the operation was the backwash. The benefits of this process were to rearrange the particles of the ionic exchanger inside the column (uniformity of packing). During the up flow backwash, the resin bed should expand in volume by 50 to 80% of pumped water for backwashing.

It was preferable to regenerate the cation exchangers with HCl solution because all its salts (chlorides) were water soluble and no precipitation occurred. Some demineralized water plants used H₂SO₄ solution to regenerate the cation exchangers but cautiously due to the above mentioned precipitation problems (CaSO₄ mainly). It was known that the exhausted cation exchangers contained high concentration of cations (mainly calcium and magnesium). Therefore, when H₂SO₄ was used as a regenerant in 2% concentration or more, precipitation occurs. This would block the column due to the pressure build up inside the column and reduce the flow rate of

Table 3. The concentration for cations and anions in water.

Ions	Input concentration (ppm)	Output concentration (ppm)
Ca ⁺⁺	200	Nil
Mg ⁺⁺	60	Nil
Na ⁺	34	0.4
K ⁺	16	0.02
Mn ⁺⁺	12	0.007
Total Hardness	750	Nil
SO ₄ ⁻	184	Nil
Cl ⁻	74.9	0.001
SiO ₂ ⁻	13.2	0.04
CO ₃ ⁻	26.1	0.102

solutions. Apart from that, it would lower the total exchange capacity of the exchanger. To overcome this problem, this might need to start with low concentration of H₂SO₄ (1.0%) which should be increased gradually to (2.5%) then to (5%) which might take more time for regeneration compared to that of HCl. In addition to that any mistake made by the operator or any damage in the acid flow meter or any fluctuation in the water or acid flow rates would cause a problem, so the operation is exposed to risk.

After the regeneration step it could be noticed that there were two rinsing steps (slow and quick rinse). The benefit of the slow rinse was to remove the remaining acid or base between resin beds. It was followed by the quick rinse. Furthermore, the experiment shows, that washing of the anion exchangers using acidic decationized water which (effluent from the cation exchanger) is advantageous in order to avoid formation of insoluble hydroxides (mainly calcium and magnesium hydroxides), and to reduce the effect of the base.

Conclusions

Usually, most of the cation exchangers were regenerated with HCl solution without any precipitation problems. However, HCl is rather an expensive solution. Apart from that it is corrosive, what makes it dangerous for the environment after its disposal.

The experiments showed that the strongly acidic cation exchanger could be regenerated by 5% sulfuric acid directly without any precipitation problems (insoluble sulfates). The regeneration time (45 min) was lower than the three consecutive concentrations of sulfuric acid (90 min). This non corrosive acid (sulfuric) was environment friendly and at the same time safe for the piping. Moreover, its low price made it competitive to hydrochloric acid for the regeneration in demineralized water plants.

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