

CHAPTER IV

EXPERIMENTAL INVESTIGATION

4.1 Experimental Apparatus

Three laboratory-scale columns were constructed and used in the experimental study. Figure 6 shows a general view of the experimental set-up, while the flow diagram of the experimental ion exchange process is shown in Figure 7. All three ion exchange columns were made up of 6.35 cm. (2½ inches) inside diameter, 120 cm. long PVC pipes, with mesh no. 60 polyethylene net placed at the top and bottom of the columns to prevent the escape of resins. Detailed drawing of the columns is presented in Figure 8. The first and third columns contained 2 cu.dm. of H-form Duolite C-20 SAC resins each while the second column was filled with 2 cu.dm. of OH-form A-102 D SBA resins. The characteristics of both Duolite resins are shown in Tables 3 and 4. A rotameter and a resistivity meter were connected to the system to measure the deionization flowrate and detect the quality of treated water, respectively.

4.2 Wastewater Used in the Study

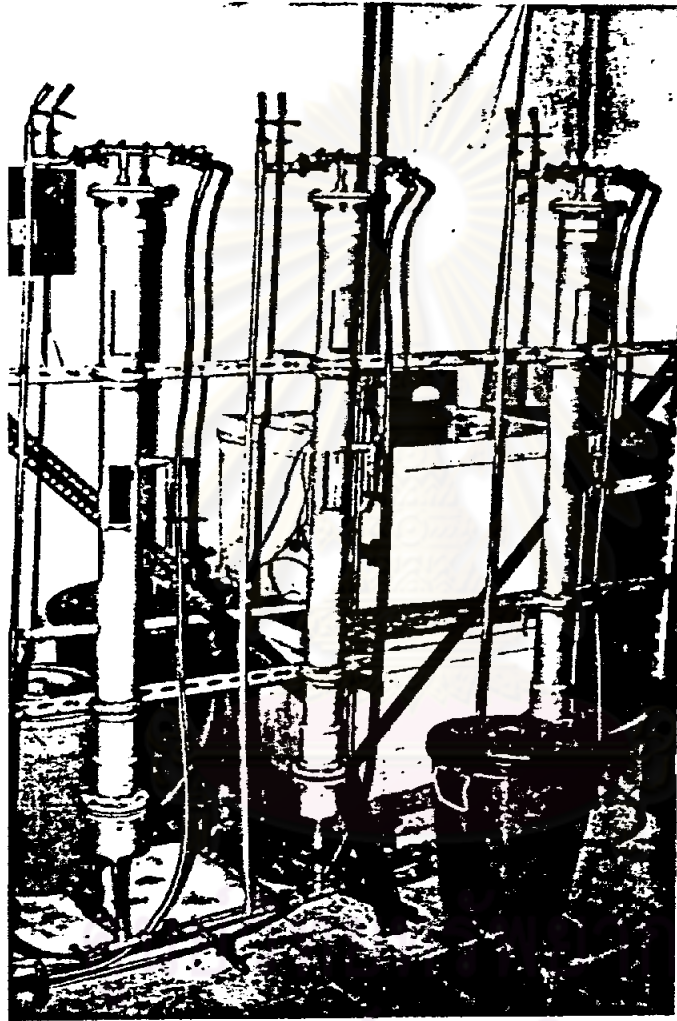
The feed water to all of the ion exchange system investigated in this research was synthetic wastewater. It was produced by simply diluting the chromic acid solution obtained from chromium plating bath of a local plating factory. This chromic acid, found to be

Table 3 Characteristics of Duolite C-20 Strong Acid Cation
Exchange Resin (13)

Property	Duolite C-20
Description	moist, translucent
Compositon	sulfonated DVB
Specific Gravity (Na form)	1.32 to 1.34
Moisture Content (%)	50-53 (H)
Bulk Density (lbs./cu.ft.)	50-52 (Na)
Effective size (mm.)	0.45-0.55
Uniformity Coefficient	1.4-1.8
Total Exchange Capacity (min.)(meg./ml)	2.0
pK.	0.0
Service pH range	0-14

Table 4 Characteristics of Duolite A-102D Strong Base Anion
Exchange Resin (14)

Property	Duolite A-102D
Chemical Classification	Strongly basic, quaternary ammonium type 2
Structure	polystyrenic
Appearance	beads, pale yellow
Net Weight as Shipped (lbs./cu.ft.)	47.5 (Cl)
True Density	1.09 (Cl)
Total Capacity (meq./ml.)	1.5 (Cl)
Size (mm.)	0.3-1.2
Service pH range	0.7



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Fig. 6 General View of the Experimental Set-up

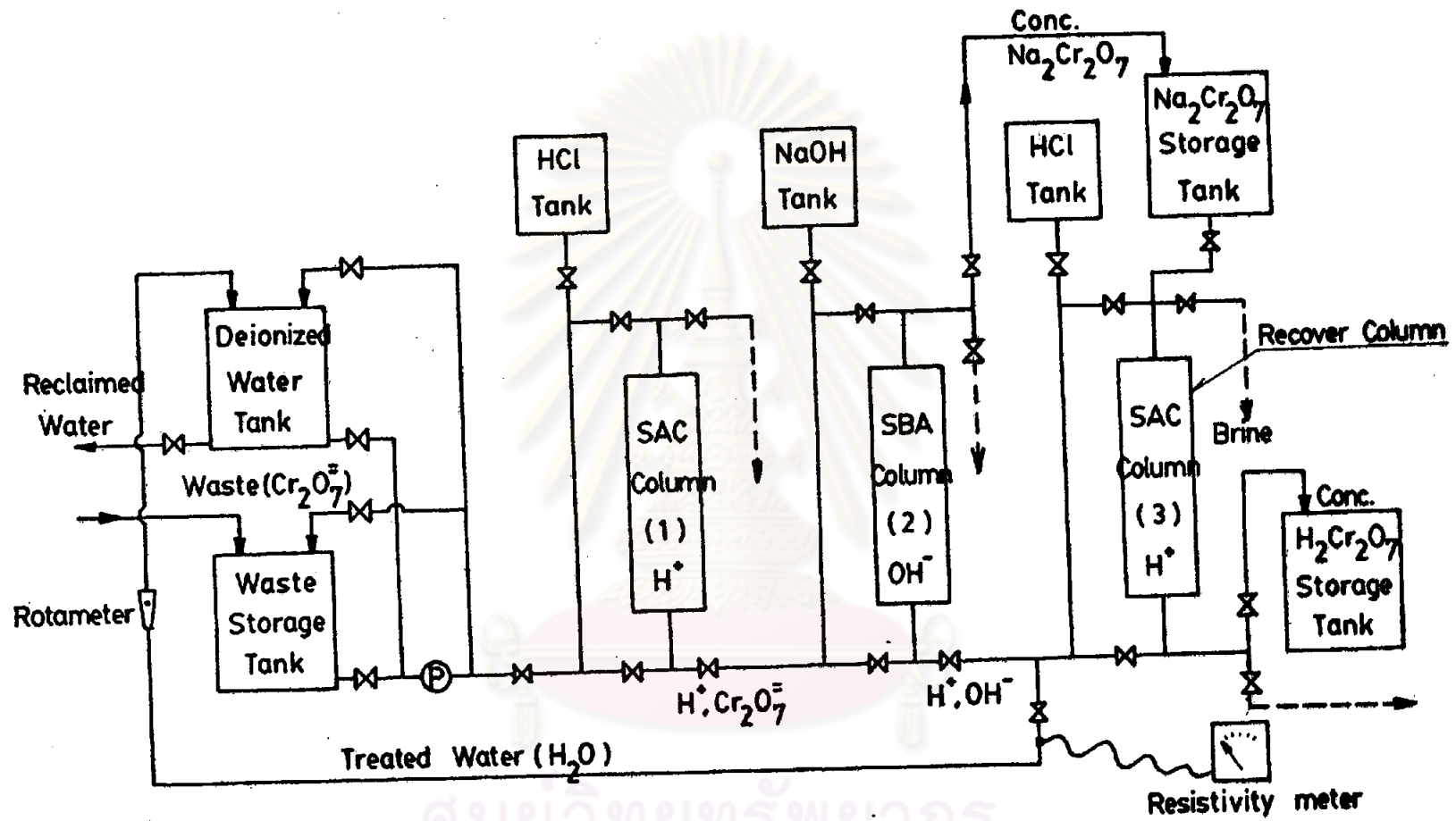


Fig. 7 Flow Diagram of the Experimental Ion Exchange Process for Chromium Reclamation

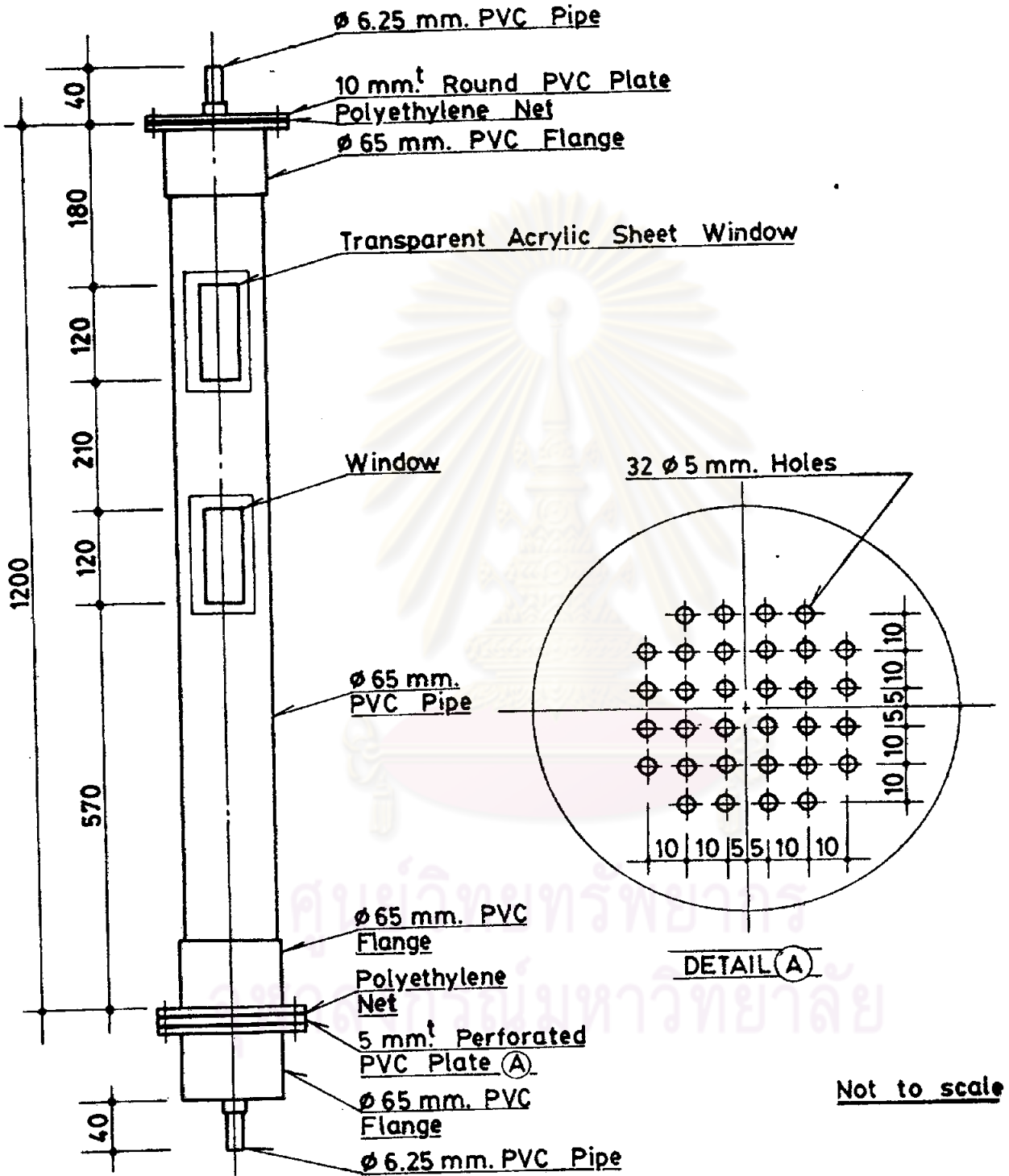


Fig. 8 Drawings of Experimental Ion Exchange Columns

about 220,000 mg/cu.dm. Cr VI concentrated, was mixed with the chromic acid recovered from the treatment unit and diluted with tap water to about 100 mg/cu.dm. Cr VI. The synthetic wastewater was stored in a 350 cu.dm. PVC tank before pumped to the ion exchange system at the downflow rate of 32 cu.dm./hour or 16 bed volumes/hour (BV/hr.). The flowrate was measured by using a rotameter and was controlled by the in-line needle valve as well as the by-pass valve. Returned wastewater through the by-pass valve, once discharged into the storage PVC tank, created sufficient turbulence to ensure the uniformity of the wastewater in the tank.

4.3 Experimental Program

In each cycle of the deionization process, the treated water with the resistivity of $20 \times 10^4 - 50 \times 10^4$ ohm-cm. (conductivity of 2-5 μ S/cm.) would be partially kept for the preparation of hydrochloric acid and sodium hydroxide regenerants and slow rinsing purposes. Another portion was stored in a 60 cu.dm. plastic container for fast rinsing purpose. The breakthrough volumes were recorded as the resistivity of the product water was reduced to as low as 0.2×10^4 ohm-cm. (conductivity of 500 micro siemen per cm.). After 600 cu.dm. or 300 BV of feed water were passed through the exchange beds and/or after breakthrough of each cycle of chromic acid recovery, the

ion exchange columns were backwashed and counter currently regenerated. After the regeneration was completed, the resins were upflow-slow rinsed with 12 cu.dm. (6 BV) of deionized water at the same flowrate as that in the regeneration process. Then, the exchange columns were further downflow fast rinsed with deionized water at the flowrate of 32 cu.dm./hr. or 16 BV/hr. for 15 minutes.

It is noteworthy that, in every cycle, the SAC columns were regenerated at the flowrate of 150 cu.cm./min.(4.5 BV/hr.) with 4 cu.dm. of 8% by weight hydrochloric acid which is equivalent to the regeneration level of 166 gm.HCl/cu.dm.resin. This is so in order to keep the condition of the feed water from the first SAC column to the SBA resins column constant for all circumstances and only the effects of the sodium hydroxide regenerant could be examined. For the recovery column, the exchange capacity of SAC resins will be the same in every cycle with this constant regeneration condition so that only the effects of feed sodium dichromate solution could be studied.

For the SBA resins column regeneration, concentration and/or quantities of sodium hydroxide regenerants were changed in each run in order to determine the optimum concentration of the sodium hydroxide regenerant so as to obtain the maximum chromic acid concentration. In the first part of the study, the regenerant volume of 4 cu.dm.(2 BV) was used while the regeneration

flowrate was kept constant at the rate of 150 cu.cm./min. (4.5 BV per hour) and variation of the sodium hydroxide regenerant concentration ranging from 3.5 to 15.0 per cent by weight was investigated. History curves of SBA column regeneration and sodium hydroxide regenerant concentrations V.S. peak & composite Cr VI values of sodium dichromate solutions curves were drawn and the optimum sodium hydroxide solution was obtained.

In the second part of study, the effects of regeneration flowrate were investigated at the optimum sodium hydroxide regenerant concentration. Four cu.dm. of 7.5 per cent by weight sodium hydroxide regenerant were used at the regeneration flowrate of 100 and 200 cu.cm./min., which were equivalent to 3.0 and 6.0 BV/hr., respectively.

The SBA resins column regeneration with different regenerant concentrations at the same regeneration level were studied in the third part. The regeneration level of 162.5 gm. NaOH/cu.dm. resin, which is the regeneration level of the optimum sodium hydroxide regenerant concentration obtained from the first part, was kept constant. The concentrations of sodium hydroxide regenerant were varied ranging from 3.5 to 15.0 per cent by weight so that the effects of contact time and regeneration level on the regeneration could be evaluated.

In the fourth part of the experiment, concentrated and diluted sodium dichromate solutions, eluted from SBA column regeneration, were separated. The concentrated sodium dichromate

solution was passed through the recovery column of SAC resins while the diluted one was used as the regenerant of the SBA resins column prior to the fresh sodium hydroxide solution. Higher concentration of chromic acid were expected to be resulted.

The sodium dichromate solution eluted from the SBA column during the regeneration was collected and stored in 20 cu.dm. plastic container. This composite sodium chromate solution was fed to the recovery SAC column, at the flowrate of 16 cu.dm./hour (8 BV/hour). This would result in concentrated chromic acid as its effluent, which could be collected and reclaimed.

4.4 Sampling and Analysis

In each experimental run, two samples of influent and five samples at 400, 500, 550, 575 and 600 cu.dm. of effluent were collected and analysed for pH, Cr VI, iron, zinc and copper. In the regeneration of SBA column, samples of the eluted sodium dichromate solution were intermittenly collected and analysed for pH and Cr VI. As a consequence, the history curves of the regeneration could be determined. The samples of eluted sodium dichromate stored in a 20 cu.dm. plastic container were taken and analysed for pH, Cr VI and sodium.

In the recovery of chromic acid, samples of the produced chromic acid were intermittenly grabbed and the composite samples

of recovered chromic acid were also collected. Both types of samples were analysed for pH, Cr VI, sodium and acidity.

The pH measurement was done through the Beckman Zeromatic SS-3 model 96 pH meter whereas sodium was detected by using the Lange's flame photometry technique. Chromium, acidity, zinc, iron and copper were tested in accordance with the Standard Methods.⁽¹⁵⁾



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