

SEPARATION OF RESIN TYPES IN MIXED BED
ION EXCHANGE COLUMNS

By

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2009

Submitted to the Faculty of the
Graduate College of the
Oklahoma State University
in partial fulfillment of
the requirements for
the Degree of
MASTER OF SCIENCE
May, 2011

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ION EXCHANGE COLUMNS

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ACKNOWLEDGMENTS

I wish to express my deepest gratitude to my advisor Dr. Gary L. Foutch. What I am today is largely due to his inspiration, kindness, understanding and invaluable guidance throughout the course of my masters program. I would like to thank Mr. William Moore from Aquatech who trained me to work on the pilot plant. I would to thank my colleague Mr. Joon Yong Lee for his assistance and help with my study. I am very grateful to Dr. Heather Fahlenkamp and Dr. Josh Ramsey for serving on the advisory committee and aiding me with the completion of this study.

I am very thankful for the funding provided by Aquatech International Corporation and Anadarko Petroleum Corp. Special thanks must be given to my parents S. Gopalakrishnan and R. Lakshmi and my fiancé S. Pavithra whose emotional support was unfailing throughout my time at Oklahoma State University. Their love and support deserve my undying gratitude.

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NOMENCLATURE

v	Velocity of the resin bead (m/s)
d	Diameter of resin bead (m)
m	Mass of the resin bead (g)
V	Volume of the resin bead (m^3)
m_f	Mass of fluid contained in the same volume as the bead (g)
g	Acceleration due to gravity (m/s^2)
ρ	Density of the resin bead (g/ml)
ρ_f	Density of the fluid (g/ml)
C_d	Drag coefficient
$\bar{\rho}$	Ratio of fluid to bead density

CHAPTER I

INTRODUCTION

Water, as we know, is indispensable to human life, playing a multitude of roles helping us sustain our day to day activities. Industries rely on water for all levels of production. Water can be used as a raw material, coolant, solvent, energy source and as a transport agent. Many modern industries require high purity water to ensure process continuity and product quality. Water of extremely high purity (ultrapure water) is required by several specific industries such as, coal and nuclear power plants, pharmaceutical and semiconductor manufacturing.

Process water with ionic impurity less than 1 $\mu\text{g}/\text{kg}$ (ppb), with corresponding low levels of particulate and microbial contaminants, is termed as high purity or ultrapure water (Sadler 1993). The impurities to be removed during its production include ionic, microbial, dissolved gases, organics, colloids and particulates. Commercially available techniques for reducing the concentration of ionic species in water include reverse osmosis, electrodialysis, ultrafiltration and ion exchange. Ion exchange plays a vital role in the manufacture of high purity water. The demand for purer water led to innovations in ion exchangers and ion exchange techniques. Mixed bed ion exchange methods use high quality synthetic resins which remove ionic impurities present in water.

The focus is now on optimization of ion exchange techniques resulting in increased productivity. Hence, modern ion exchange processes need to meet the demands for higher quality water at low cost and with minimum waste. Resin separation in mixed bed ion exchange is a vital step during the regeneration process and determines the extent of sodium and sulfate or chloride into the system from caustic and acid regenerants, respectively. The objectives of this study are:

- a) Identify the effect of two variables: backwash flowrate and bed expansion on the cross contamination of resin types during separation.
- b) Compare cross contamination for two different forms of resin, the H/OH form and Na/SO₄ form.
- c) Investigate the efficiency of interface isolation methods for resin transfer utilizing a pilot scale plant.

ION EXCHANGE

Ion exchange is a branch of separation science which deals with the partition of charged species between different system regions (Townsend 1993). Ion-exchange is defined as the reversible stoichiometric interchange of ions between a solid phase (the ion-exchanger) and a solution; the ion exchanger is usually insoluble in the medium in which exchange occurs. Ion exchange is now a firmly established unit operation and an extremely valuable alternative to other operations such as adsorption, distillation and filtration. Though used in several chemical processes, ion exchange finds applications in three broad categories: removal of ions, substitution and separation. Ion exchange can effectively remove sodium, chloride, calcium, magnesium and other ions to produce large volumes of ultrapure water required by food, power, microelectronics and pharmaceutical industries.

MIXED BED ION EXCHANGE

In a mixed bed ion exchange column, cation and anion resins are adjacent through mixture. In H-OH form the cation resin contains exchangeable H^+ ions and anion resin contains exchangeable OH^- ions. Consequently, even better purification is obtained than with the application of an alternating sequence of cation and anion exchangers because exchanges are coupled by water dissociation equilibrium of the H^+ and OH^- ions to water (Dorfner 1972). Hence, a mixed bed ion exchange column represents a powerful single unit for demineralizing water; and with refinement, produces water of utmost purity.

NEED FOR EFFECTIVE RESIN SEPARATION

As water is continuously passed through the mixed bed, the cation and anion resins remove the impure ions and release equivalent ions back into the water. In a normal H-OH form operation the cation resin releases H^+ ions and the anion exchanger releases OH^- ions. Eventually the ion exchange resin will saturate and their ability to exchange ions gradually decreases and leads to bed exhaustion. When this condition is reached the resins have to be regenerated to their original forms so that they can be used again. Anion resin is regenerated with caustic (NaOH) and cation resin is regenerated with a suitable mineral acid such as sulfuric or hydrochloric acid.

The most important step in mixed bed regeneration is resin separation. Improper separation leads to cross contamination of the resin. Some of the cation resin, for example, will contaminate the anion resin layer. Since the anion resin is subsequently regenerated with caustic, the entrapped cation resin in the anion resin is completely saturated with sodium ions. Similarly, when some of the anion resin contaminates the cation resin, the entrapped anion resin is completely saturated with sulfate or chloride ions depending on the acid used for cation resin regeneration.

Cross-contamination of resins leads inevitably to equilibrium leakage of sodium and sulfur/chloride ions into the treated water. Figure 1 shows initial equilibrium leakage from a mixed bed as a function of the initial ionic loading (Grimshaw and Harland 1975). The initial loading is the fraction of sites in the sodium or chloride form on the cationic or anionic resins, respectively.

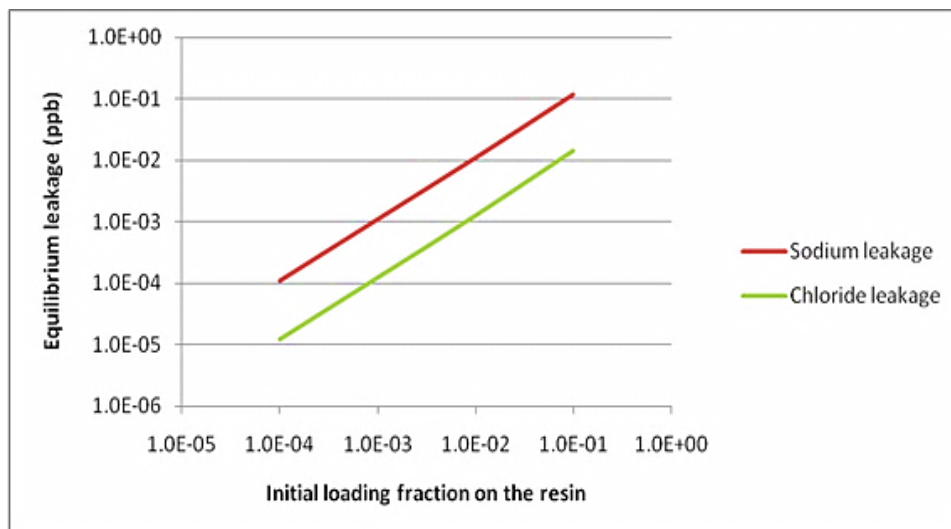


Figure 1. Predicted equilibrium leakage from Dowex 650C and 550A (Yi and Foutch 2004)

This could be from beads caught in the other resin prior to regeneration, or regeneration efficiency as a result of insufficient chemical treatment or poor rinsing. The chloride curve is lower in equilibrium leakage than the sodium curve because of the higher anionic selectivity coefficient for chloride to hydroxide on the resin. Figure 1 indicates that if 1% of the exchange sites are in the sodium form (99% in the hydrogen form) then the concentration from the bed initially will be about 0.01 parts-per-billion. For 0.1% cross-contamination the initial sodium leakage from the service cycle should be about 1.0 parts-per-trillion. For chloride a 1% cross-contamination of anionic beads in the cation treated with hydrochloric acid regenerate will yield an initial leakage of about 1.1 parts-per-trillion. If sulfuric acid is the cationic regenerant, a 1% cross-contamination would result in significantly lower leakage because of the higher preference of divalent ions by the anionic exchange resin. This ignores the sulfate that may originate from potential cationic degradation.

For ordinary deionization, minute contamination is of little consequence. However, for applications like condensate polishing where one or two parts per billion (ppb) can be serious, cross contamination of resins cannot be tolerated. Mixed bed resins operated in the ammonia form leak 100 to 1000 times higher levels of impurities than the same resins operated in the hydrogen cycle (Webb and Larkin 1996). Hence the target for allowable cross contamination in ammonium form mixed bed is as low as 0.08% (Sadler 2001). Therefore, if the purity of water from mixed beds is to be maintained at ultra-low contaminant concentrations it is essential that the ion exchange resins are separated completely.

CHAPTER II

RESIN SEPARATION IN MIXED BEDS

EARLY DEVELOPMENT

The first recorded use of mixed cation and anion exchangers consisted of weakly acidic and basic resins that were non-regenerable (Martin 1952). The first use of strongly acidic and basic exchangers was reported by McGarvey and Kunin (1951) and was called a mono bed as the initial aim was to replace the two bed deionization with a single bed system. The anion resin was separated from the cation resin and transferred to a separate vessel for regeneration. The cation resin was regenerated in the service vessel. After regeneration the anion resin was again transferred back to the service vessel. A second patent, in 1954, described a mixed bed process where regeneration of both types of resin took place within the service vessel (McGarvey and Kunin 1954).

It was soon found that the concept of single mixed bed replacing a two bed system required more frequent regeneration and higher operating costs. There were problems with precipitation of insoluble metal hydroxides during regeneration. These issues led to the approach of using a two bed cation-anion exchange system followed by a mixed bed.

The mixed bed removed the trace impurities leaking from the two bed system. It received a relatively small ionic load and had less frequent regeneration. Majority of the plants then adopted the cation-anion exchange plus the mixed bed principle, although some installations with high quality raw water adopted a mixed bed only approach.

CONDENSATE POLISHING

Throughout the power, utility and chemical industries, large volumes of water are evaporated and condensed to either separate the solvent from solute, act as a driving force for turbines and for many other purposes. In most cases the condensates are recycled. Although these condensates are distilled waters of relatively high purity, in many instances they must be purified before they can be reused. Feed water specifications of a modern advanced high pressure steam raising plant are so low that direct re-use of condensed steam from turbines is unacceptable. The condensate which may be contaminated with cooling water and corrosion products from the steam generator, the turbine, the preheater or the pipelines as a result of leaks in the tube bundles, must be purified before it is used again. Hence the condensate needs to be demineralized or 'polished' by mixed bed ion exchange techniques employing resins. This technique is mainly advantageous since it permits very high flow rates.

The water purity required from boiler feed water make-up plants is a conductivity of $< 0.15-0.20 \mu\text{S}/\text{cm}$ and silicate concentrations of $< 0.02 \text{ ppm}$ (Dorfner 1972). Most of these plants employ mixed bed systems containing a mixture of strong acid cation resin and strong base anion resin. The resin is in service vessels which treat the condensate at a

relatively high specific flow rate as higher rates improve filtration and reduces the number of vessels and amount of resin required.

Because of the high condensate flow rates, condensate polishing plants operate at much higher throughputs than normal make up water treatment units. At such high flow rates, the kinetics of ion exchange resins gives satisfactory operation but as the resins age, their performance deteriorates. This leads to organic foulants in the mixed bed that affects performance. The resins used today in condensate polishing are typically produced from styrene-divinylbenzene (SDVB) copolymers. The SDVB copolymer consists of polystyrene chains with divinylbenzene cross linking between the styrene chains. The SDVB copolymer based ion exchange resin has the advantage over most other synthetic polymers from a stability and capacity standpoint (Webb and Larkin 1996). Present day manufacturing techniques allow physically stronger resins to be produced with tailored and very consistent particle sizes. There is now a widespread use of Uniform Particle Size (UPS) resins in condensate polishing with benefits being seen in the separation process (Shields et al. 2006) and, consequently, in the quality of the final water.

Condensate polishing requires properly designed equipment, properly selected resin and well trained operators. A comprehensive surveillance and monitoring program is also required to maximize potential benefits of the whole operation. Condensate polishing has found extensive application especially in the nuclear and power industry, where there is demand for high purity water. Although advances have been made to produce greater water quality there still remains a need to economize the process and increase efficiency. An important step to achieve this high efficiency is the effective separation of resins prior to regeneration.

RESIN SEPARATION TECHNIQUES

A wide range of methods are available for the effective separation of anion and cation resin prior to regeneration. The methods vary from simple to complicated processes and are chosen depending on the separation desired, economics, and spatial constraints.

CONVENTIONAL APPROACH: DENSITY SEPARATION

The majority of mixed bed separation processes available rely on the slightly different falling speeds of anion and cation resins in water. Resin mixture in the separation vessel is backwashed into suspension and allowed to settle. The denser cation resin beads settle more quickly than the anion resin beads. An effective separation is generally achieved with an anion resin upper layer and a cation resin lower layer. The interface consists of a mixture of both cation and anion resin beads. The top anion layer is transferred hydraulically via a side take-off port to an anion regeneration vessel. Resins are regenerated separately, returned and mixed before rinsing. This method was initially developed by McGarvey and Kunin after which improvements were done to the whole process design for better resin separation and transfer (McGarvey and Kunin 1951).

Another conventional method was to regenerate both the cation and anion resin in the same vessel after the density separation. A regenerant collector/distributor is located at the interface for respective regenerants (McGarvey and Kunin 1954) however, it is seldom used today. A well operated and maintained conventional regeneration system may not achieve much better than 2 - 5 volume % cation in anion resin and 5 - 10 volume % anion in cation resin after separation and transfer (Sadler 1986). However, with

customized operating procedures, it is possible to reduce cross-contamination to 0.4% anion in cation resin and 0.5% cation in anion resin (Crone 1987).

INTERFACE ISOLATION METHOD

The interface isolation approach was a non-proprietary technique developed in the early 1970's. In this method, the anion and cation are separated by their density similar to initial steps of the conventional approach. As shown in Figure 2, after separation, the anion resin is transferred hydraulically to an anion regeneration tank. The interfacial layer containing a mixture of both resins is removed to an isolation vessel via a second take-off set just below the interface whilst the pure cation resin remains in the vessel (Hattori 1989), (Kusano and Nawata 1987).

The separated cation and anion resins are then regenerated using the appropriate regenerants. This significantly minimizes cross contamination of anion in cation and cation in anion. The interface is isolated till the next batch of spent resin arrives for regeneration. This system requires control over the height of interface, that is, over the resin volumes in each charge.

INERT RESINS

The use of inert resins having an intermediate density between that of anion and cation resins was introduced in the year 1976 (Shields et al. 2006). These inert resins formed a distinct buffer layer between the upper anion resin layer and the lower cation resin layer. This minimizes the cross contamination of anion and cation resins at the interface. These beads need to be ionically neutral and chemically and physically inert to the classifying fluids and the regenerants.

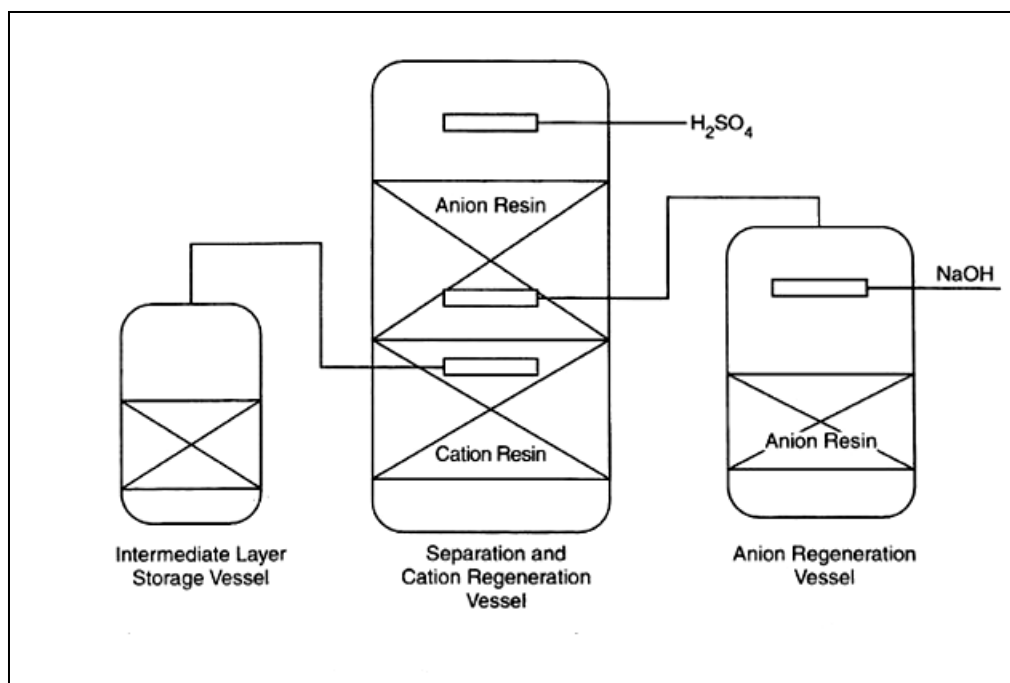


Figure 2. Interface isolation method (Kusano and Nawata 1987)

The inert beads also had to impart a different conductivity than the anion and cation beads so that the conductivity sensors used in certain separation systems could detect them (Lefevre and Sato 1981). Soon after its advent, inert beads with enhanced qualities such as better stability, wettability, uniformity, better strength and hydraulic stability were developed (Chonde 1985; Osei-Gyimah 1988).

Recently, as field experience was gained and better separation methods developed, interest in these resins decreased. They are known to foul and attract bubbles, causing problems during handling and transfer. The advent of Uniform Particle Size resins, which allowed excellent separation, also led to the phasing out of inert resins.

CONESEP SEPARATION SYSTEM

This high efficiency mixed bed separation system was first described in 1977 with a full scale plant at Aghada, ESB, Ireland in 1980. The system consists of a vessel for resin separation/anion regeneration, a cation regeneration vessel and a resin isolation vessel as shown in Figure 3. Exhausted mixed bed resins are initially transferred to the resin separation/anion regeneration vessel which has a conical bottom section. They are cleaned and separated by backwashing in the usual manner. The lower cation resin layer is transferred to the cation regeneration vessel via a transfer pipe drawing from the base of the conical bottom section. To aid this transfer, a flow of water is introduced through the porous base of the Conesep unit with part of this flow bled from the top of the vessel and the remainder acting as resin motive water. The slight upflow helps to keep the resins fluidized, stabilizing the cation/anion interface. As the cation resin is transferred, the cation/anion interface gradually descends through the vessel into the conical section and eventually enters the transfer pipe without any disturbance. A sharp resin interface passes along the transfer pipe where it is detected by sensors, either by conductivity or optical methods, and transfer into the cation regeneration vessel is stopped. The small quantities of cation and anion resin held in the transfer line then directed to the interface isolation vessel and combined with the next exhausted mixed bed charge entering the Conesep system for regeneration. To achieve the highest separation efficiency, the anion resin is regenerated with sodium hydroxide and then given a second separation by backwashing. This takes advantage of the fact that any cation resin entrained in the anion resin will now be in the denser sodium form and the anion resins in less dense hydroxide form.

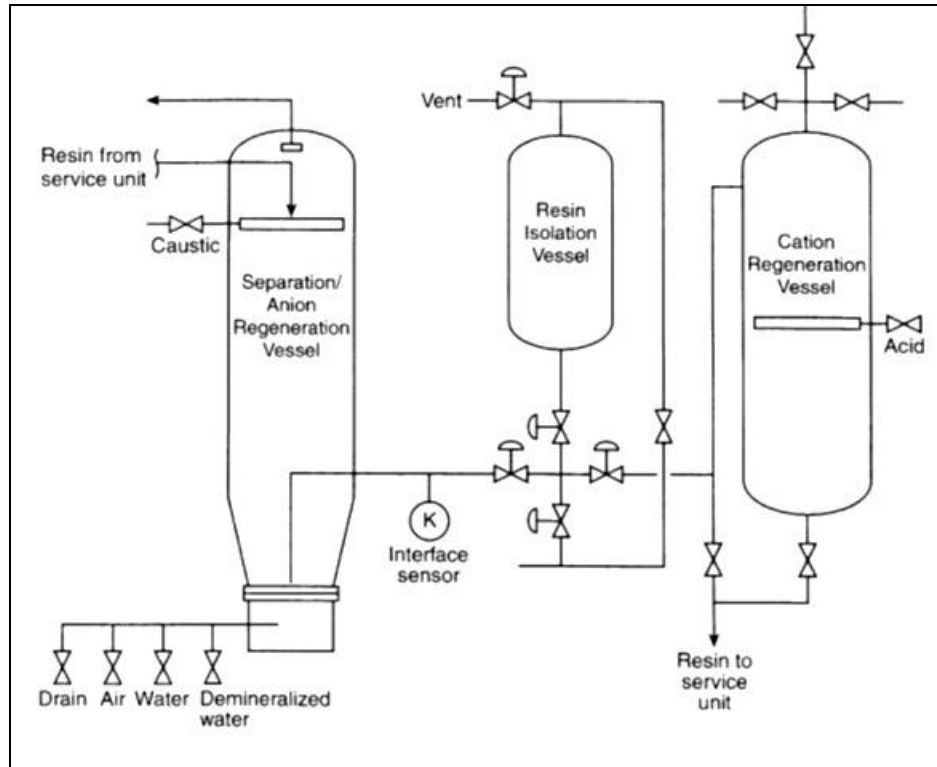


Figure 3. Conesep mixed bed separation system (Emmet and Grainger 1979)

The very small volume of cation resin thus recovered is again directed to the interface isolation vessel. Cation in anion resin cross contamination levels of about 0.05 to 0.07 volume % are usually obtained (EPRI 1997). A significant advantage of this method is that the bottom transfer systems do not depend on the separated cation/anion interface being at a precise height in the vessel. Thus operators have the freedom to adjust resin ratios to suit their needs (Emmet 1977).

FULLSEP SEPARATION SYSTEM

Southern California Edison Company San Onofre Nuclear Generating Station developed a condensate polishing system similar to the Conesep process during 1983 (Shields et al.

2006). The system is described in Figure 4. A cation-mixed bed arrangement was chosen with an innovative design for separation/regeneration.

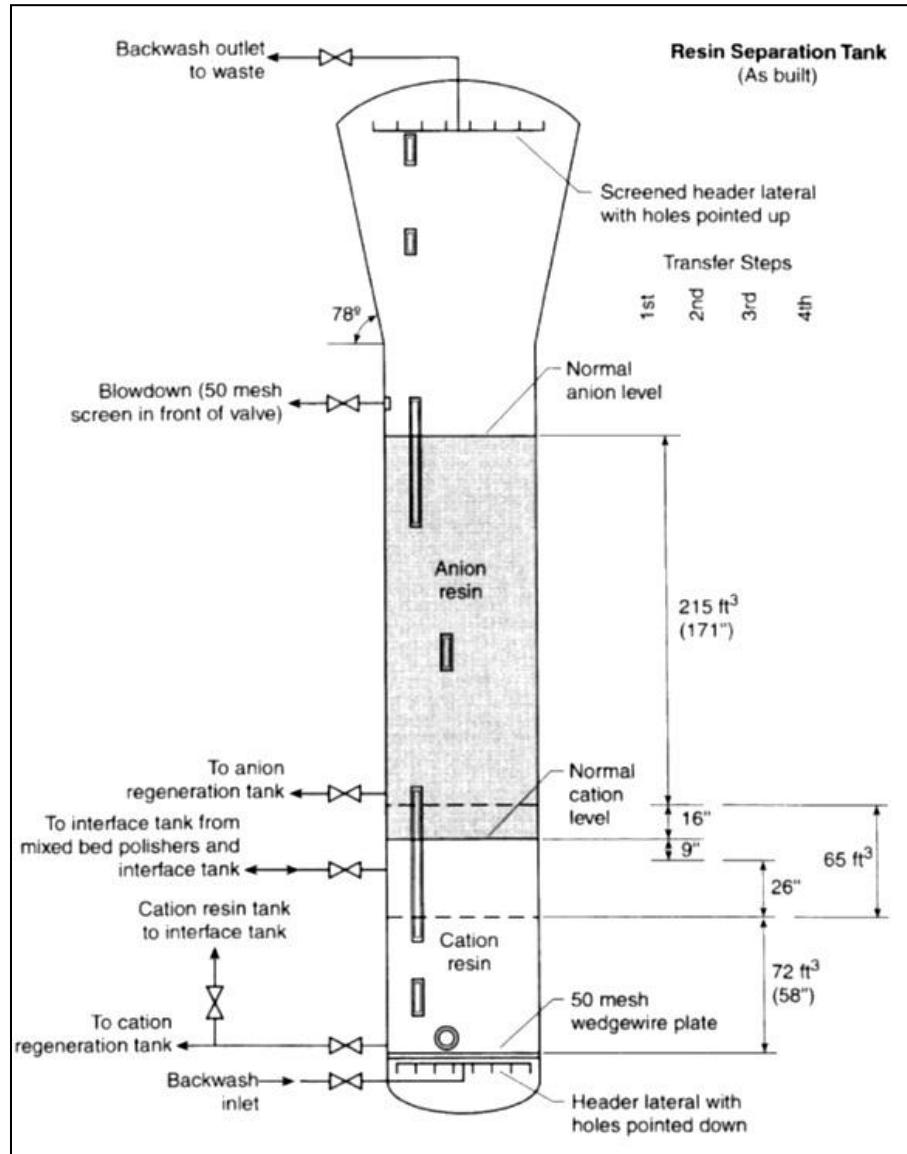


Figure 4. Fullsep separation system (Auerswald 1987)

It employs both interface isolation and bottom transfer of cation resin in the same process (Auerswald 1983). The system was first commissioned in September, 1985. The San Onofre mixed bed separation system has proved to be very effective, yielding both low

cation in anion resin levels as well as low anion in cation resin levels (Auerswald 1987). The design was adopted commercially and has been supplied to many power stations.

SEPREX PROCESS

This proprietary separation improvement technique, also called the caustic floatation process, was developed by the Graver Water Division of the Graver Company (EPRI 1997). In this method, the exhausted cation and anion resin are initially separated into two distinct layers by backwashing similar to the conventional separation system. The anion resin layer is drawn off and transferred to the anion regeneration vessel along with the cation resin present in the interfacial layer. The anion resin along with the cationic contaminants is regenerated with concentrated solution of sodium hydroxide (16 - 20 % by weight).

The solution has a density such that the small volume of entrained cation resin sinks to the bottom of the vessel while the anion resin floats on the top. The floated, regenerated anion resin is transferred to the resin storage vessel with the caustic solution and rinsed before combining with regenerated cation resin. The sodium form cation resin in the anion regeneration vessel is transferred to the cation regeneration vessel where it is regenerated with the next batch of resin. A schematic representation of Seprex process is shown in figure 5. The removal of most of the cross contaminating cation resin leads to a useful reduction in the overall sodium levels in the final mixed bed (Olijar and Salem 1970).

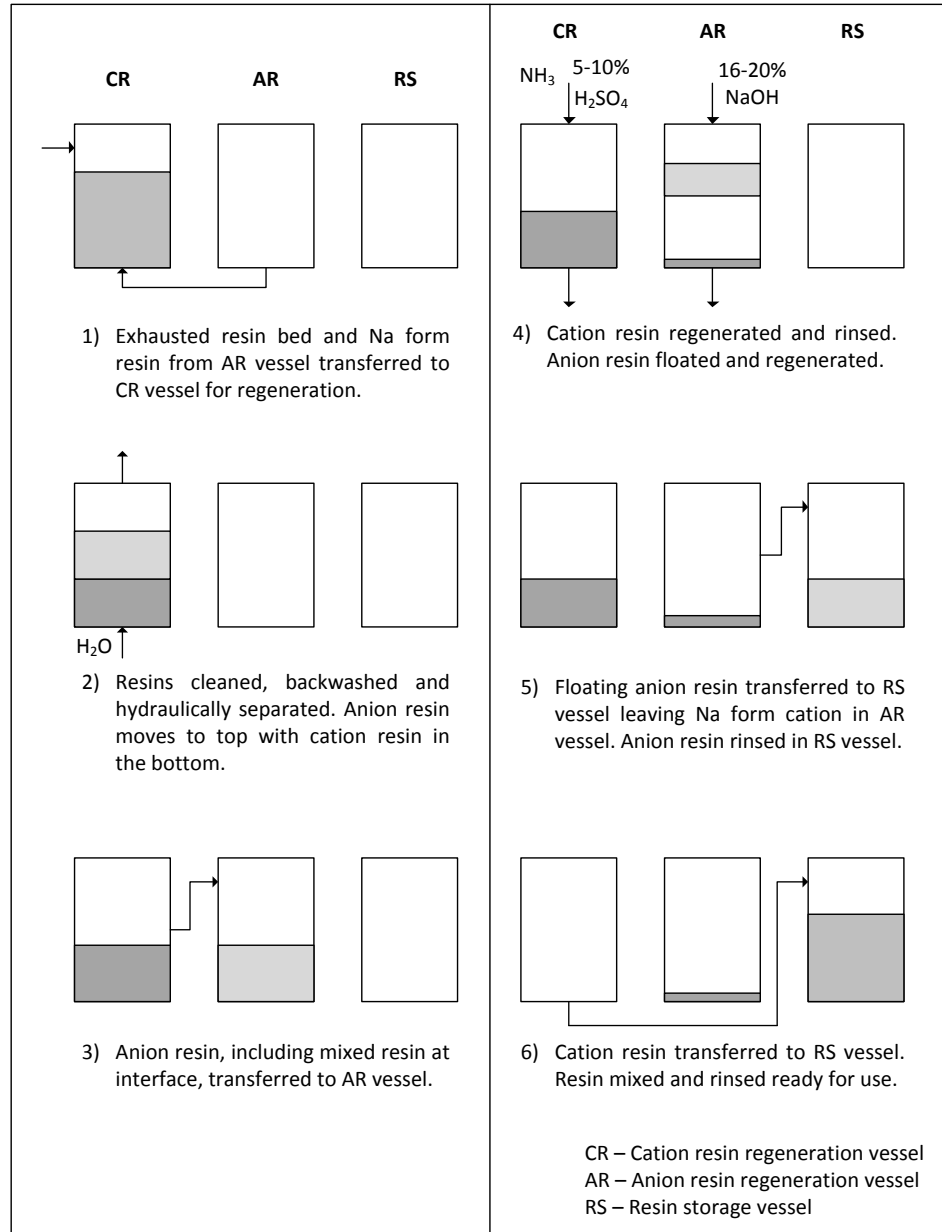


Figure 5. Seprex process (Webb and Larkin 1996)

SEPRA EIGHT PROCESS

This system, introduced in 1983, combines the techniques employed in the Seprex and Conesep processes (Salem and Scheerer 1983). System operation through the bottom transfer of cation resin to the cation regeneration vessel follows the Conesep procedures. However, inert resin is also used in the Conesep separation vessel to minimize anion

resin contamination of the cation resin. The cation resin is regenerated in the conventional manner with 4 -10% by weight sulfuric acid. The anion, inert and small amount of entrained cation resin are left in the separation vessel.

The anion resin is regenerated and floated to the anion resin rinse vessel as in the Seprex process. After rinsing the regenerated cation and anion resins, the anion resin is transferred to the cation regeneration vessel where the conventional air mixing and mixed bed rinse are performed.

RESIN ON RESIN APPROACH

This procedure originated in South Africa in the mid 1970's (Sadler and Bolton 1996). It was originally designed to reduce the problem of enhanced sodium leakage arising from the level of cross contamination from conventional mixed bed separation systems where top anion resin layer is drawn off to a separate vessel. However, it is now applied to systems where cation resin is separated and transferred. Resins are first separated as effectively as possible and the anion and cation resin is transferred to their respective regeneration vessel. The anion resin, containing traces of cation resin, is regenerated and rinsed the normal way. Any entrained cation resin is fully converted to the sodium form in this process. The regenerated anion resin, containing the entrained cation resin, is transferred back to the cation regeneration vessel and air mixed with the cation resin that is still awaiting regeneration. The remixed resin is allowed to stand with periodic air mixing then separated. The cation resin is regenerated and rinsed in the normal way before being recombined with the anion resin for a final rinse before service.

This non-proprietary procedure reduces the sodium contamination of the final mixed bed. Its use requires that the regeneration plant has the necessary connections to allow the separated and regenerated anion resin to be remixed with the cation resin. Since its advent, this method has been modified and customized to suit the particular location and to increase separation efficiency. For example, the resin on resin approach coupled with the interface isolation method showed significant improvement in separation efficiency (Sadler 1995). One fossil power station estimates that it reduces the sodium originating from cross contamination by a factor of 10 thus correcting for less than perfect separation (McCarthy and O'Connor 1992). The procedure has been adopted by many PWRs to allow them to achieve very low sodium leakage when operating in the H-OH form. Reports from these stations are positive with a typical comment being that it has allowed them to reduce their sodium values to an acceptable level with essentially zero cost with the exception of a bit of time.

RESIN CLEANING, SIZING AND SEPARATION SYSTEM

In this procedure, there is a steady feed of resins to be cleaned, sized and separated. As shown in Figure 6, the exhausted mixed bed resin feed is initially passed through a pre-treatment section where crud and other impurities are removed. The resin is fed into a column of water flowing upwards in a specially designed vessel. The flow of water in this vessel is carefully regulated in such a way that the anion resins alone are swept upwards. The heavier cation resins sink to the bottom of the vessel. The anion resins overflow into the anion resin settling section and into an anion regeneration vessel while the cation resins are moved to a cation regeneration vessel. The remaining cation resin is transferred to the cation regeneration vessel.

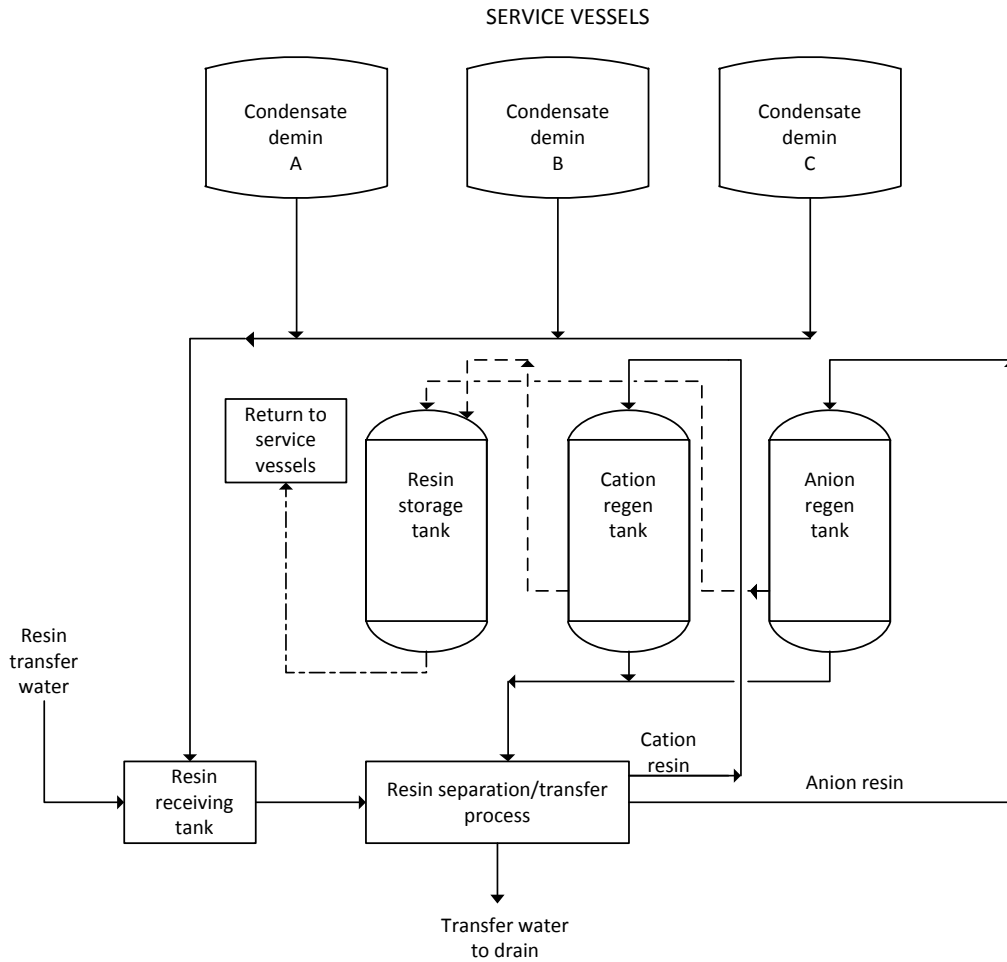


Figure 6. Resin cleaning, sizing and separation system (Shields et al. 2006)

AMSEP PROCESS

The Amsep process is based on the density differences between anion and cation resin in the sulfate and ammonium (or other amine) forms, respectively. The first step in this process is to air scrub and backwash the resin. This removes some iron from the surface of the resin and facilitates resin separation to upper anion and lower cation. It prevents magnesium precipitation from occurring within or on the anion resin during ammonium sulfate regeneration.

The second step is to regenerate the separated resin using 2% ammonium sulfate as SO_4 . This regeneration completes the conversion of cation into the ammonium form and removes the exchanged calcium, magnesium and sodium. It also begins the conversion of the anion resin into the sulfate form. Following the 2% ammonium sulfate regeneration step, 6% ammonium sulfate is introduced into the resin in an upward flow. This step completes the conversion of the anion into the sulfate form and generally results in the liberation of carbon dioxide from the anion resin. The upward flow during this step separates the cation and anion resins while not affected by the liberation of carbon dioxide gas. By introducing the ammonium sulfate in steps, efficient and effective stripping of chloride and sodium from the resin is accomplished. The final step is to float the anion resin out of the separation tank and into the anion regeneration tank using 30% ammonium sulfate. Ammonium sulfate is transferred from the anion regeneration tank to the ammonium sulfate measuring tank thereby minimizing its consumption. 30% ammonium sulfate solution has a density intermediate the cation and anion resins allowing this flotation to occur readily at 60°F (EPRI 1997). At higher temperatures, a slightly higher concentration of ammonium sulfate is needed to float the anion resin. Following transfer, the cation resin is sluiced to the cation regeneration tank and both resins are rinsed, air scrubbed and backwashed. The cation resin is regenerated with sulfuric acid and the anion resin with caustic followed by a short ammonia rinse. This process offers an advantage of cleaning the resin beads thoroughly in addition to the near complete separation achieved. Reports of its performance at the supercritical Mohave Power Station show that the Amsep process is very effective (Auerswald and Cutler 1991; Meyers 1993).

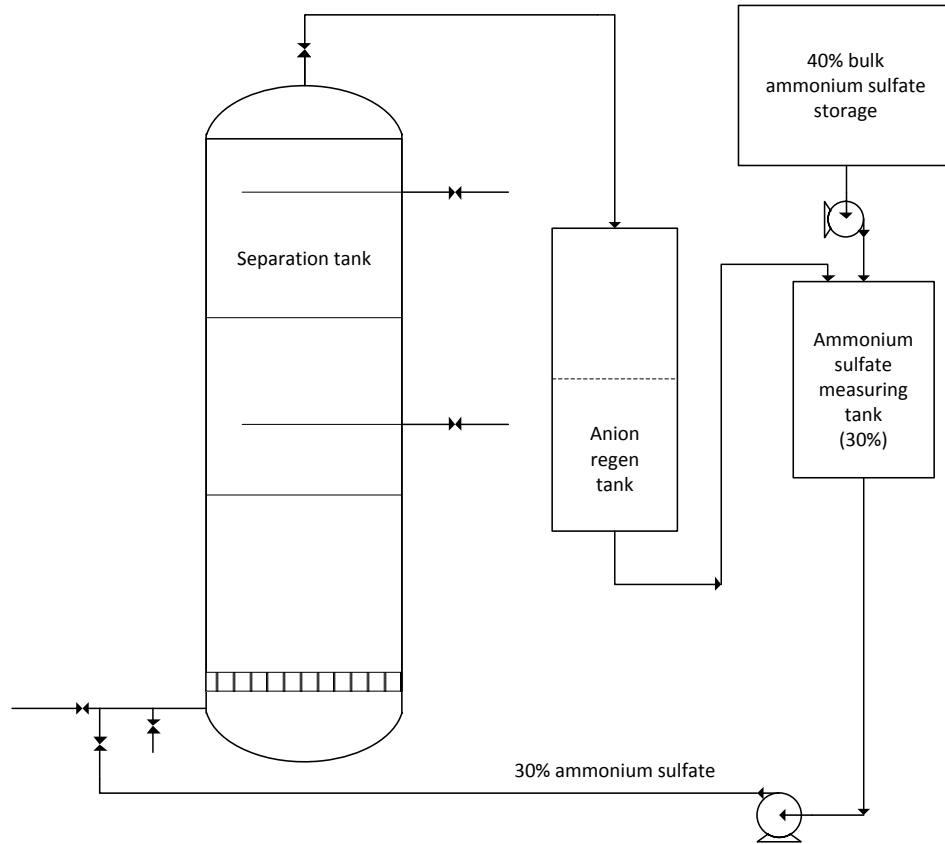


Figure 7. Amsep separation process (Webb and Larkin 1996)

EFFICIENT AND EFFECTIVE RESIN SEPARATION

Choosing the right separation method depends on the purity of water desired, spatial availability, manpower and economic constraints. Production of high purity water needs greater separation efficiency which requires use of advanced separation techniques. Operators need to be well trained to perform the process effectively. Customization and modification of processes are sometimes necessary to meet spatial and economic limitations. Conventional regeneration systems with appropriate customization can reduce cross contamination to about 0.4% anion in cation resin and 0.5% cation in anion resin (Crone 1987). Utilization of inert resin beads can reduce cross contamination of

cation in anion resin from 0.2 – 2% depending on the care with which separations are performed (Bevan 1989). The Conesep system can achieve cross contamination levels of <0.1% cation in anion resin and <0.5% anion in cation resin (Sadler 1986). A “tall thin” separation tank with interface tank can limit cross contamination to 0.03% cation in anion and 0.15% anion in cation (Auerswald 1986). Utilization of Resin cleaning and sizing system can reduce resin cross contamination to 1 – 2% range for both phases of separated resins (Stengel and Pillow 1990). The Amsep process can achieve a separation efficiency of 0.01% cation in anion and 0.01% anion in cation (Auerswald and Cutler 1991).

CHAPTER III

GRAVITY SEPARATION OF RESINS

INTRODUCTION

Irrespective of the resin separation technique used prior to regeneration, a mixed bed has to be differentiated into two distinct layers of anion and cation resin before transfer. This is achieved by the gravity separation method which makes use of repeated backwash and settling steps. Resin separation is directly proportional to the degree of fluidization of the bed. The degree of fluidization can be affected by the velocity of the backwash water, duration of flow and bed expansion during isothermal conditions. During backwashing, the DI water enters from the bottom of the service vessel at a set flow rate thereby fluidizing the resin bed and causing it to expand. Although there is a sharp difference in particle density between the two types of resin, there still remain small quantities of resin intermixing both below and above the interface. It is these quantities of cross-contamination that will be evaluated in this study.

DESCRIPTION OF EXPERIMENTAL EQUIPMENT

The experimental unit is a full system containing three 12 inch columns, a large feed tank and all required plumbing for multiple operations. Water can be fed from either the top or

bottom of each vessel and water and resin can be transferred among all three columns. Air can be fed to all three vessels as required for specific operations. The service column has six sampling valves spaced six inches apart that allow both sampling and resin transfer above and below an interface. A 440 V pump moves water through the system. Sensors for conductivity and pH are connected to sampling lines that can be moved to multiple locations. The DI water feed tank includes a heater to operate the system at any steady state temperature and a level controller to prevent equipment problems. A more detailed description of the experimental unit can be found in appendix A. The Cation resin used was DOWEX 650C and the anion resin was DOWEX 550A in a 2:1 volume ratio.

ESTIMATION OF CROSS CONTAMINATION

For each trial a backwash flow rate and volume expansion percentage were selected and five backwashing steps were performed. At low flow rates it is also possible to operate for an extended time at the maximum bed expansion. However, higher flow rates can result in moving resin out of the top of the column. Initially an air mix is used to homogenize the bed prior to backwash. Ten minutes of air mixing was used prior to each experiment. After each backwash, the interface height was recorded and water was drained from the bed to allow it to stabilize within the column. Based on the geometry and the amount of resin present, the samples were obtained from the ports immediately above and below the interface. The samples were drawn from the take-off ports and then processed to estimate the weight fraction of anion resin in the cation layer and cation resin in the anion layer. Samples were placed in 20% NaCl where beads separate by density difference. This step converts the anion and cation beads to the chloride and

sodium forms, respectively. Anion beads were siphoned off. All resins were placed on filter paper, drained, rinsed and dried. Rinsing ensured that free salt was removed that might interfere with an accurate weight of small masses. The filter paper and resin were weighed, the resin removed and the filter paper reweighed and subtracted. Any salt accumulated on the filters was included in the tare weight so that the weight recorded was only for whole beads.

BACKWASH RESULTS: H/OH FORM

The initial set of experiments was performed with the cation resin in hydrogen form and the anion resin in the hydroxide form. Eight experiments were repeated with a fixed flowrate and bed expansion height. The first two experiments provided a chance to practice operating the equipment and fine tune the experimental procedure. The flowrate used for the first two experiments was 27 gpm (100% rotameter range) with a bed expansion of 100%. This was too high a flow rate for optimum performance and resulted in high cross contamination. The next flowrate chosen was 13.5 gpm (50% rotameter) and the bed was allowed to expand to 100%.

The results for 13.5 gpm flow and 100% bed expansion are shown in Figures 8 and 9. From these data the anionic beads in the cation layer controls the separation process since the fraction of anionic resin decreased with each of the five successive washes. Error bars were added considering a maximum deviation of 0.001 g during measurement. The error bars are prominently visible for the anion layer due to relatively better separation achieved from the very first backwash. Cationic resin beads in the anionic phase did not indicate a correlation with the number of washes. This could be due to some cationic

beads being pushed up along with the with anion resin as a result of high flow. However, all values were less than 0.35%, even after the first wash. A single wash showed 7% anionic resin in cation layer. The third wash resulted in less than 1% anionic beads. The fifth wash showed 4 cationic beads in an 8.667 g sample. The Dowex MSDS sheet indicates that a backwash rate of 8 gpm should be sufficient for a 100% anionic bed volume expansion. The observed values agreed with this assessment since increased turbulence was clearly noted at higher flowrates.

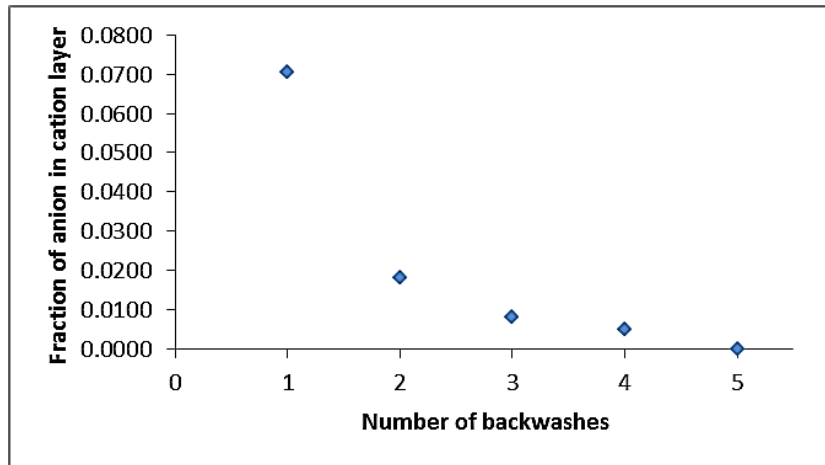


Figure 8. Fraction of anionic beads in cation layer at 13.5 gpm and 100% expansion

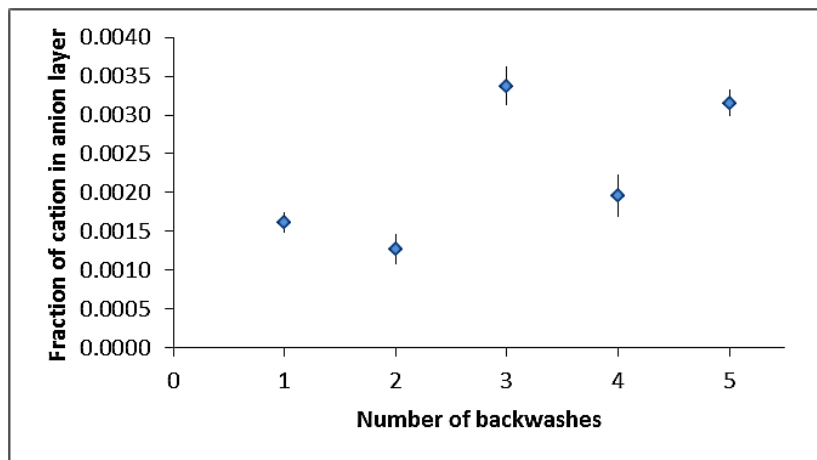


Figure 9. Fraction of cationic beads in anion layer at 13.5 gpm and 100% expansion

The next experiment used a flowrate of 6.5 gpm and 100% bed expansion and the results are shown in Figures 10 and 11. The fraction of anionic resin in the cation is lower overall as compared to Figures 8 and 9, although the fifth backwash is not as low, indicating the variance in the experimental data or the abilities to sample the column. Results indicate that 0.2% can be achieved after the third wash and maintained through subsequent washes. The cation in anionic resin range was only slightly improved with three of five washes achieved less than 0.15% while only one wash achieved this level at the higher flow rate.

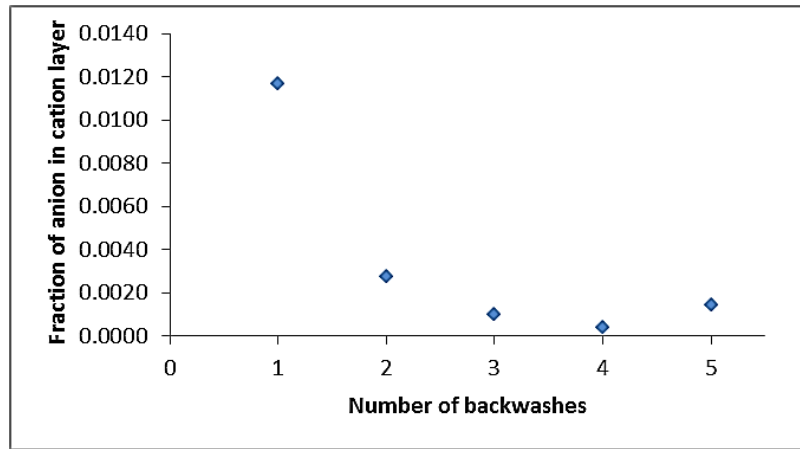


Figure 10. Fraction of anionic beads in cation layer at 6.5 gpm and 100% expansion

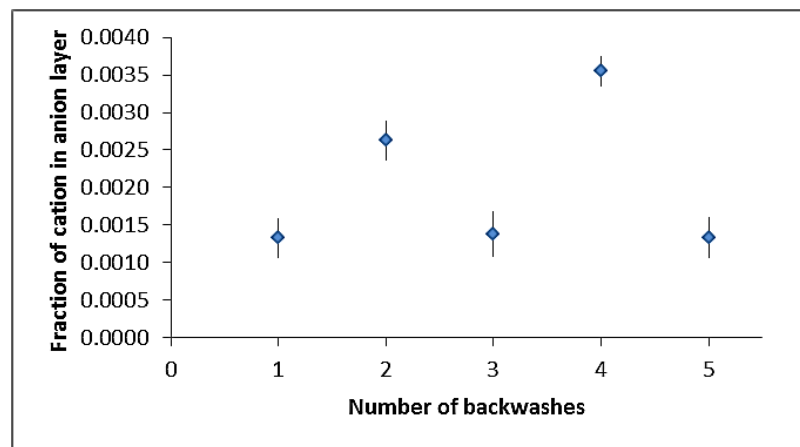


Figure 11. Fraction of cationic beads in anion layer at 6.5 gpm and 100% expansion

Figures 12 and 13 are results obtained at 4 gpm and 50% bed expansion. The fraction of anionic resin in the cation is significantly improved. The low flow and duration of expansion was not sufficient to effectively raise the anion beads from the cation layer. However, the fraction of cationic resin in the anion shows that four out of five backwashes have less than 0.3% cross contamination.

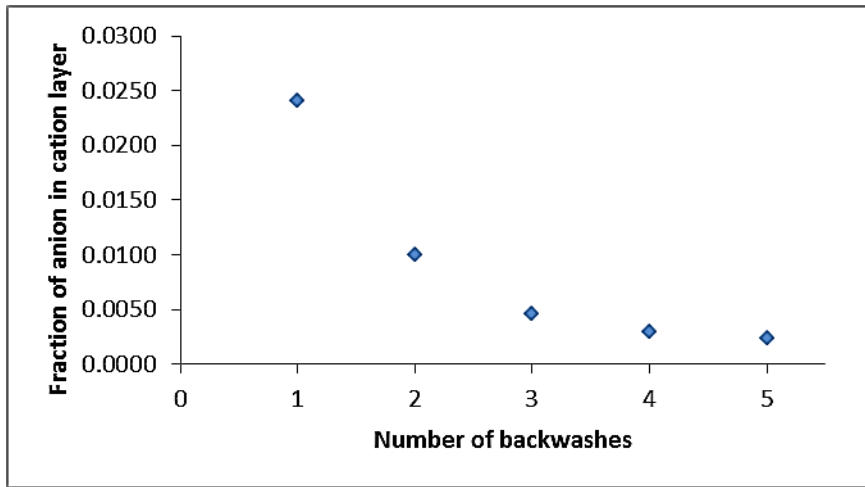


Figure 12. Fraction of anionic beads in cation layer at 4 gpm and 50% expansion

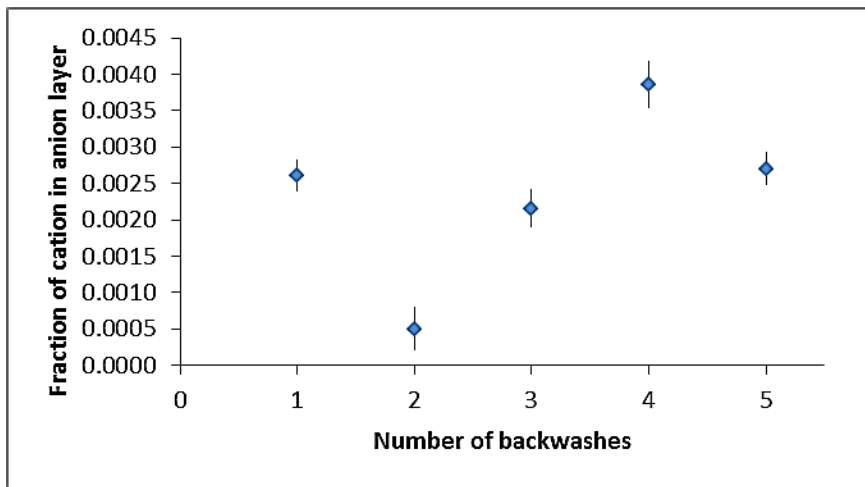


Figure 13. Fraction of cationic beads in anion layer at 4 gpm and 50% expansion

Figures 14 and 15 are results obtained at 13.5 gpm and 150% bed expansion. The results for anion layer weren't as good as expected. To achieve a greater bed expansion, a higher flow rate is required which in turn has been noted to cause more turbulence between the anion and interface layer. The minimum flow needed to achieve 150% expansion was observed to be 13.5 gpm. A higher flow rate produces significantly more mixing that lifts the cationic beads higher into the vessel. When flow is stopped the 50% additional settling height is not sufficient to compensate for the increased mixing effect.

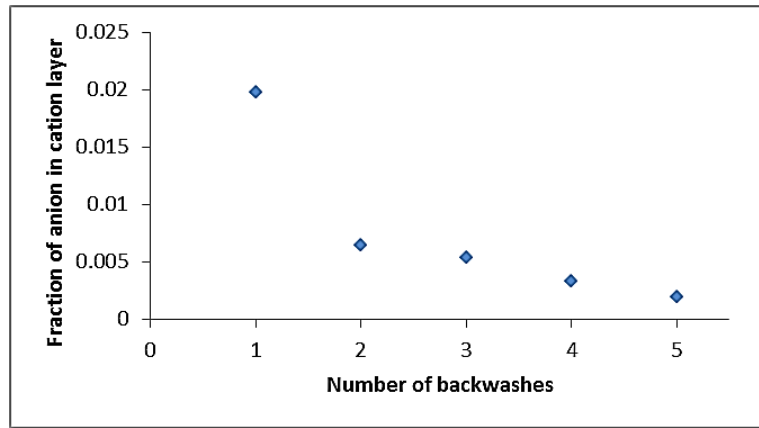


Figure 14. Fraction of anionic beads in cation layer at 13.5 gpm and 150% expansion

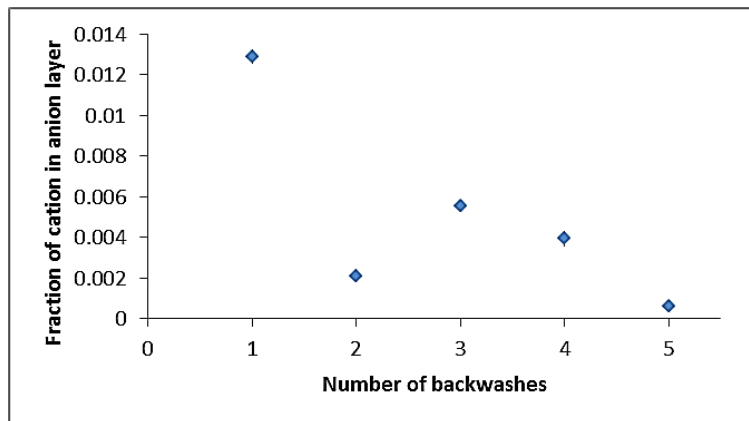


Figure 15. Fraction of cationic beads in anion layer at 13.5 gpm and 150% expansion

Figures 16 and 17 present results obtained at 6.5 gpm and 75% expansion. A flowrate of 4 gpm was not sufficient for 75% bed expansion. After five backwashes, the fraction of cation resin in anion was 0.04% and the anion fraction in cation was 0.2%. There is steady decrease in cross contamination after repeated backwash at the same conditions. The data indicate that greater bed expansion gives better resin separation at the same flow rate. The cation in anion contamination is significantly low, whereas the anion in cation contamination is higher than that for 100% backwash expansion with the same flowrate.

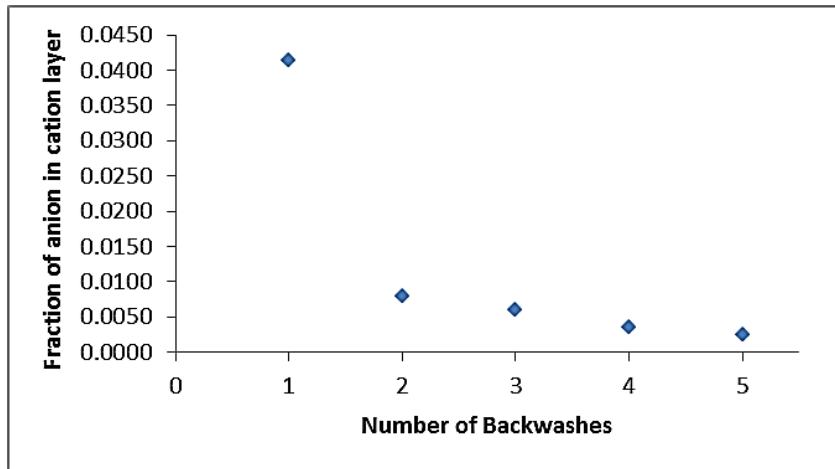


Figure 16. Fraction of anionic beads in cation layer at 6.5 gpm and 75% expansion

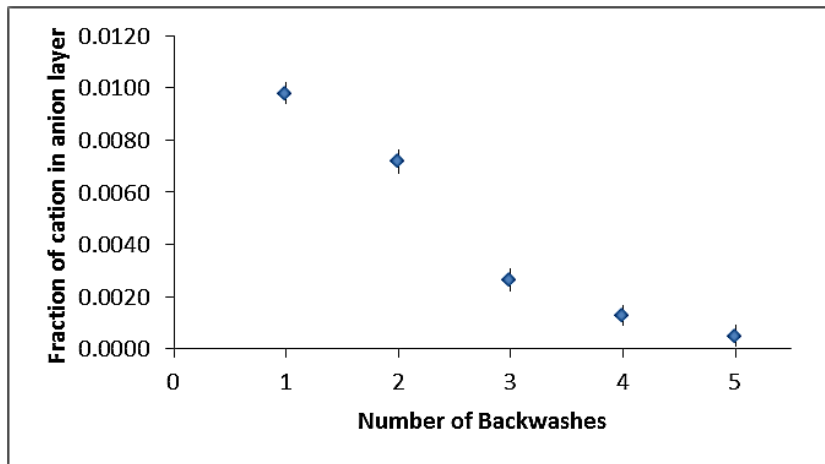


Figure 17. Fraction of cationic beads in anion layer at 6.5 gpm and 75% expansion

Figures 18 and 19 are results obtained at 13.5 gpm and 75% expansion. The higher flowrate causes increased mixing at the interface resulting in variation of cross contamination values with successive backwashes. The bed expansion is not sufficient to compensate for the increased mixing.

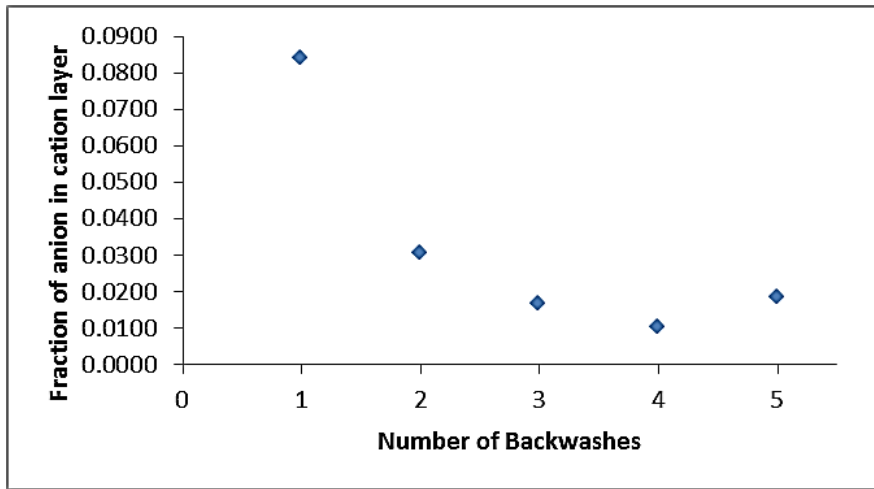


Figure 18. Fraction of anionic beads in cation layer at 13.5 gpm and 75% expansion

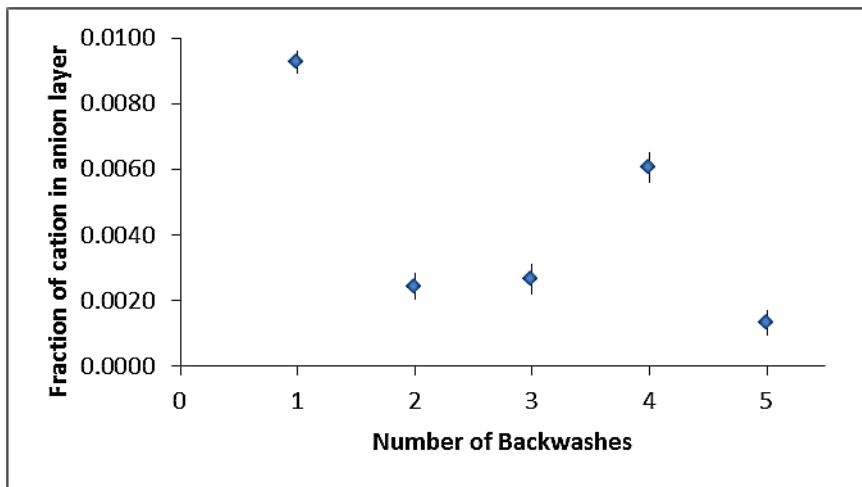


Figure 19. Fraction of cationic beads in anion layer at 13.5 gpm and 75% expansion

In order to more easily visualize comparisons with the data in Figures 8 through 19, combined charts were prepared and are presented. The three types of comparisons

available from these data are: high flow (13.5 gpm) with different backwash expansion; low flow (6.5 gpm and 4 gpm) with different backwash expansion; and 100% expansion at different flows (13.5 gpm and 6.5 gpm). Figures 20 and 21 give the results for three different expansions at a flowrate of 13.5 gpm. Figure 20 does show improvement in the anionic resin within the cationic resin layer, particularly for the first two backwashes. However, during the same experiments the amount of cationic resin in the anionic fraction is marginally worse; as indicated in Figure 21.

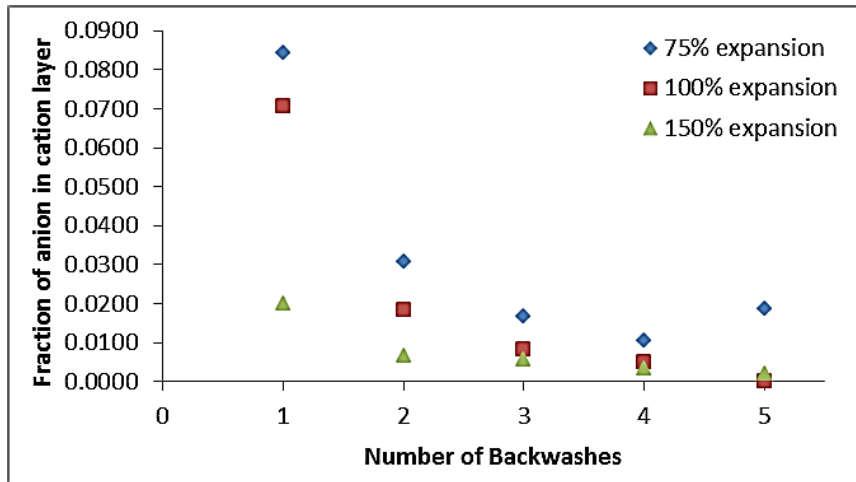


Figure 20. Differences in anionic fraction within cation layer at 13.5 gpm flow

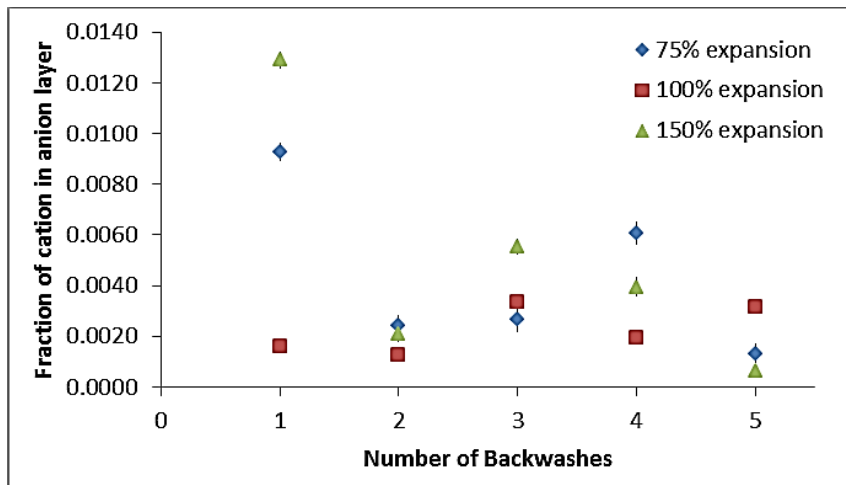


Figure 21. Differences in cationic fraction within anion layer at 13.5 gpm flow

Comparison of three different bed expansions (50%, 75% and 100%) at low flow rates are shown in figures 22 and 23. Figure 22 shows a clear preference for 100% backwash for anionic separation from the cation layer.

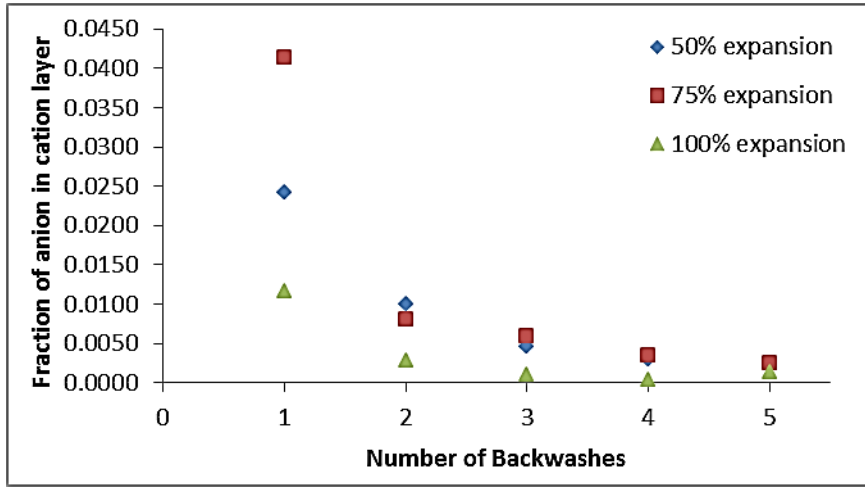


Figure 22. Differences in anionic fraction within cation layer at low flow

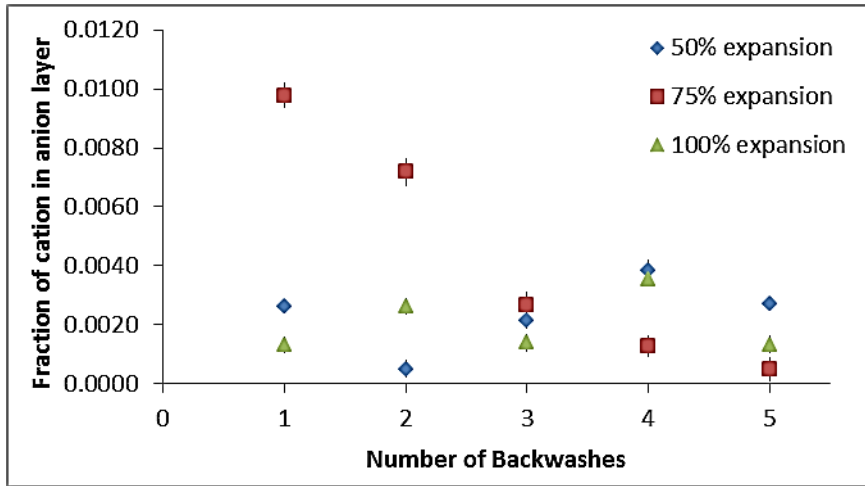


Figure 23. Differences in cationic fraction within anion layer at low flow

For the effects of the backwash flowrate with the same bed expansion (Figures 24 and 25) we can see a preference for the lower flow for the anionic resin fraction in the cation resin layer. The low flow experiment gave the lowest measurable anionic resin value of

0.1%. For cationic resin in the anion layer the average improvement at the lower flow is about 10%, with 3 of 5 values less than 0.15%.

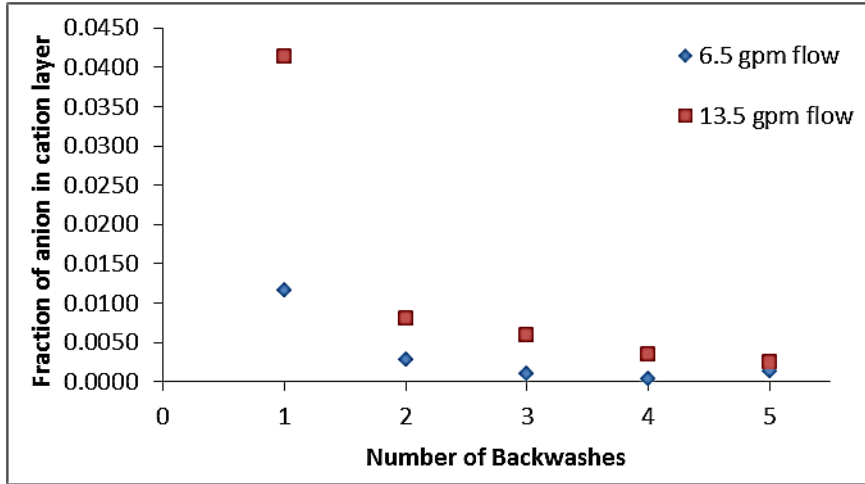


Figure 24. Differences in fraction of anionic resin in the cation layer with flow for 100% expansion.

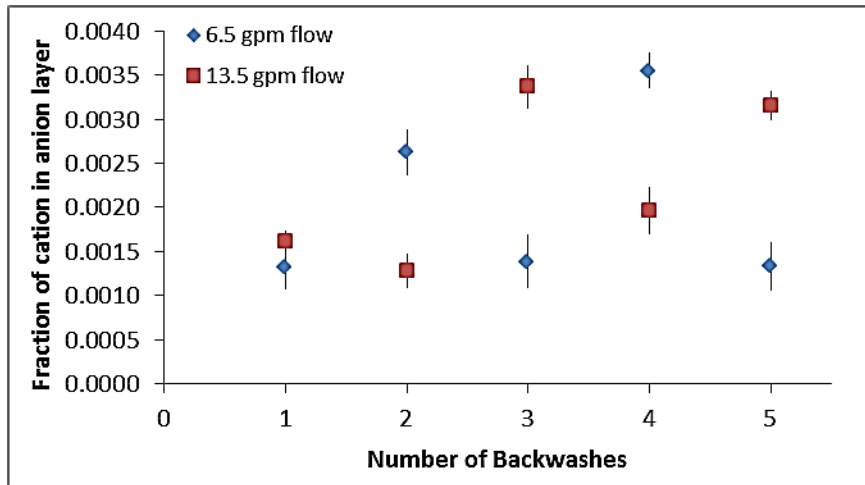


Figure 25. Differences in fraction of cationic resin in the anion layer with flow for 100% expansion.

STEADY STATE BED EXPANSION

In this experiment the bed was allowed to expand to its maximum height for a set flow rate and the steady state expansion was maintained for a fixed period of time. The bed was then allowed to settle and samples were drawn to measure the cross contamination in each layers. The intent was to define what duration of steady-state expansion was equivalent to performing 5 backwashes with the same flowrate. A flow rate of 4 gpm was chosen for this experiment. The bed expanded by 58% and the steady state expanded height could be maintained for 2 minutes 30 seconds. The fraction of cation in anion resin was found to be 0.27% and the fraction of anion in cation was 0.78%. After five backwashes at 4 gpm and 50% expansion, the fraction of cation in anion was 0.27%, similar to the steady state expansion; whereas the fraction of anion to cation was 0.23% which is significantly lower than the steady state value. The disadvantage of using the steady state expansion method for separation is that lower flow rates do not push out the anion resins from the cation layer effectively, whereas higher flowrates result in lesser steady state expansion times.

EFFECT OF WATER TEMPERATURE ON CROSS CONTAMINATION

Viscosity and density of water changes when there is a change in water temperature. This change should affect the rise and settling of resin beads. In order to investigate effect of temperature change an experiment was conducted with the DI water at 40°C and compared with results from normal experimental conditions (18°C). From the NIST data for properties of liquid water, it is observed that the dynamic viscosity of water decreases

by 34% and density decreases by 0.6% when the temperature increases from 18°C to 40°C. The results for experiments at 75% expansion and flowrate of 6.5 gpm for both temperatures are shown in Figures 26 and 27. The fraction of anionic resin in cation after fifth backwash at 40°C was 67% lower than at 18°C. This indicates lesser resistance experienced by the resin beads due to elevated water temperature which pushes out the lighter anion resin from the cation layer easily during backwash. The fraction of cation resin in anion layer, however, remains the same for both experiments at 0.05%.

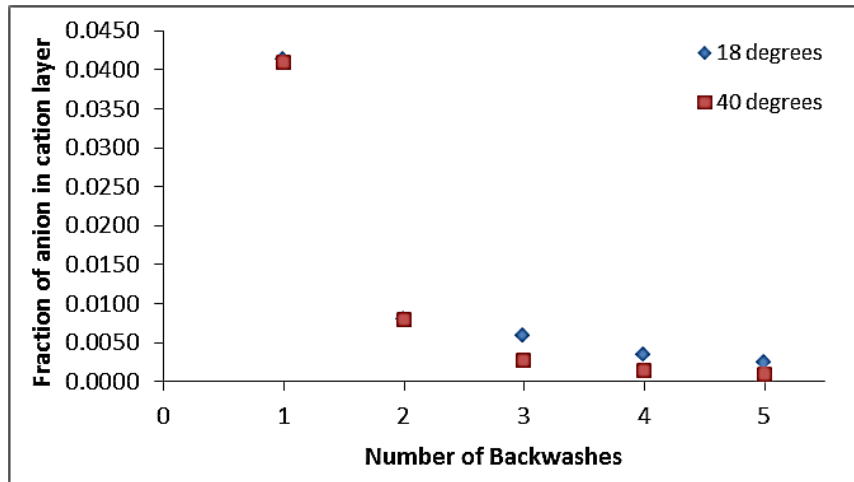


Figure 26. Differences in fraction of anionic resin in the cation layer at 18°C and 40°C

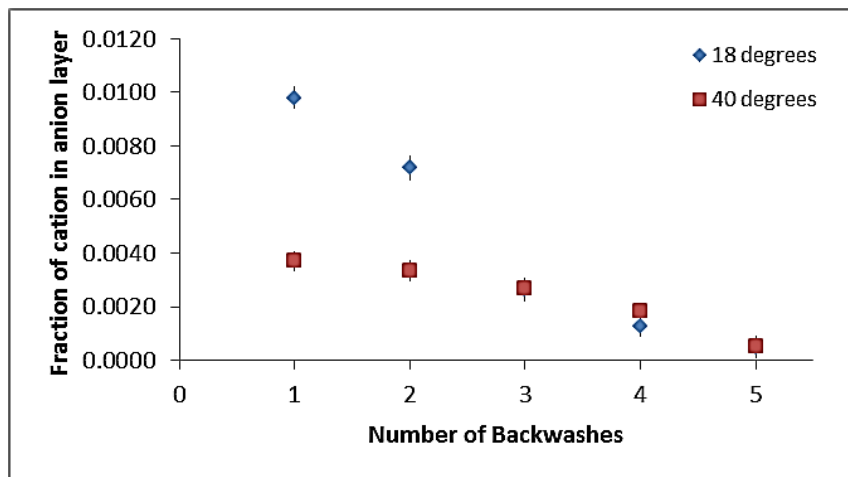


Figure 27. Differences in fraction of cationic resin in the anion layer at 18°C and 40°C

For separation of anion resin from the cation layer, low values of cross contaminations are noticed for the first two backwashes itself. This could be due to the faster settling of cation beads from the anion layer due to lesser resistance as the bed is allowed to settle.

A steady state experiment was also carried out at 40°C at a flow rate of 4 gpm. The bed expanded by 44% which was lesser compared to 58% at 18°C. The expanded steady state could be maintained for 2.15 minutes. As shown in Table 1, the fraction of anionic resin in cation layer is much higher at 40°C. This is due to the reduced bed expansion at elevated temperature. The fraction of cationic resin in anion layer, however, is lower than at 18°C due to the ease of settling during the steady expanded state. Since only one test was performed, results should be considered inconclusive.

Table 1. Effect of temperature on steady state bed expansion

Temperature (°C)	Resin fraction	
	Cation layer	Anion layer
40	0.0137	0.0007
18	0.0078	0.0027

BACKWASH RESULTS: Na/SO₄ FORM

With continuous use of a mixed bed ion exchange column, the resins are slowly depleted of H⁺ and OH⁻ ions. Ion exchange of impurities in water will eventually exhaust the resin capacity sufficiently that resin regeneration is required. In order to compare the changes in separation efficiency between fully loaded resins and resins in their pure form, gravity separation experiments were repeated with the cation and anion resins converted to sodium and sulfate forms, respectively. The two factors that affect separation in the converted form are density and resin bead size. Cation resin shrinks by 6-7% and anion resin by 13-15% in their converted forms (Applebaum 1968). The shrinkage is accompanied by increase in density. The effect of these physical changes on the cross contamination after separation was investigated.

The entire volume of cation resin was converted to sodium form by soaking the resin in sodium hydroxide solution containing an equivalent amount of sodium ions for an hour. Similarly the anion resin was soaked in sulfuric acid solution containing an equivalent amount of sulfate ions. The resin beds were rinsed with DI water until any excess ions were removed from the beds. Both resins were returned to the same column after conversion. All six separation experiments with the resins in H/OH forms were repeated in the converted forms.

Figures 28 and 29 show the comparison of cross contamination between H/OH and Na/SO₄ form resin for a flow rate of 13.5 gpm and 100% expansion. After five backwashes, the converted form has lower cation cross contamination of 0.07% than the 0.3% observed in the H/OH form. However, for the anion layer, only two of five

backwashes were less than 0.5% for the converted form resin, while the H/OH form had less than 0.5% cation fraction for all five backwashes. The cation layer has more anion in the Na/SO₄ form for all five backwashes. Increased turbulence was noticed in cation layer for Na/SO₄ form resin at a flow rate for 13.5 gpm. Though cross contamination gradually decreased with each backwash, Na/SO₄ form had an anion fraction of 1.5% in the cation layer; which was ten times higher than the value in H/OH form.

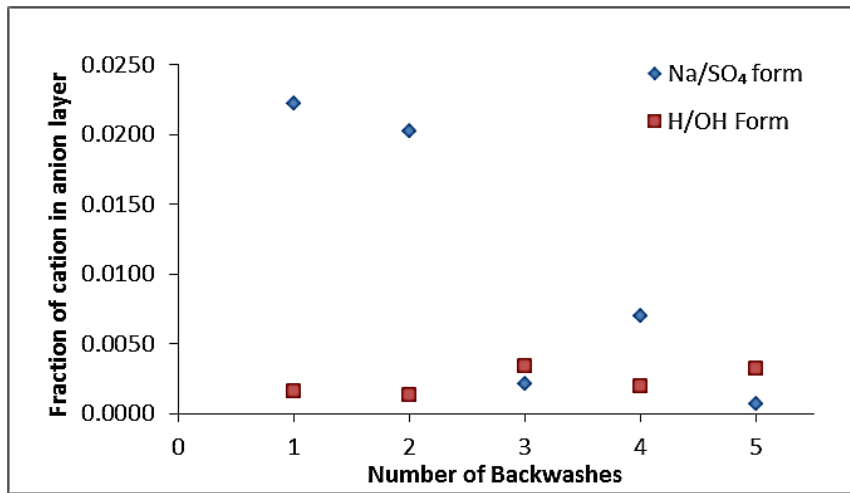


Figure 28. Comparison of cationic fraction at 13.5 gpm and 100% expansion

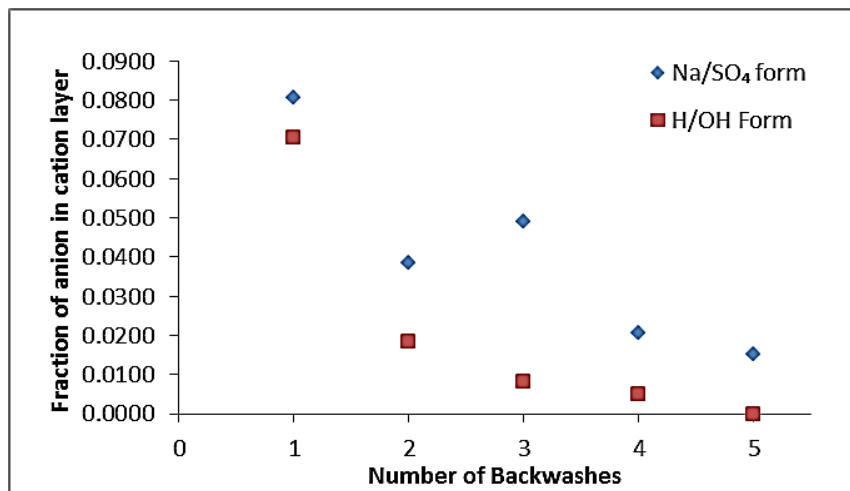


Figure 29. Comparison of anionic fraction at 13.5 gpm and 100% expansion

Results for 13.5 gpm and 75% bed expansion are shown in Figures 30 and 31. The anionic layer cross contamination values are erratic for both forms of resin due to high flow and reduced expansion. However, at the end of five backwashes, Na/SO₄ form shows lower cross contamination of 0.09% compared to H/OH form (1.32%). The error bars here are not big enough to explain a specific trend. So, there is more error than measurement alone anticipated. The effect of increased mixing is also noticed in the cation layer results for Na/SO₄ form. Na/SO₄ form has a higher anionic fraction of 2.3% compared to 1.8% in H/OH form.

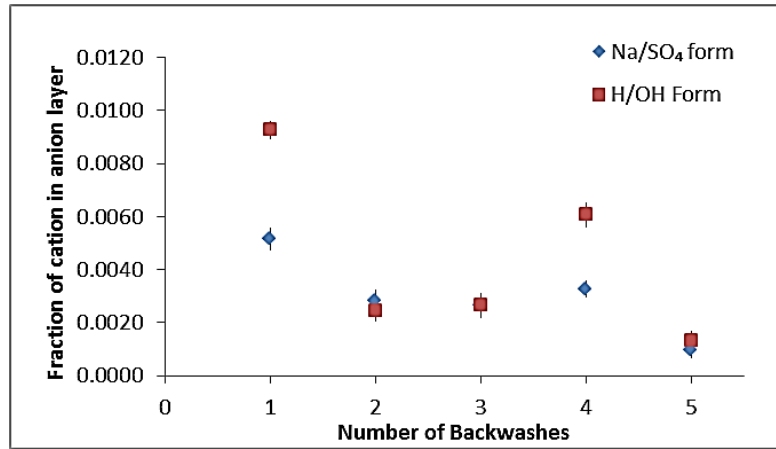


Figure 30. Comparison of cationic fraction at 13.5 gpm and 75% expansion

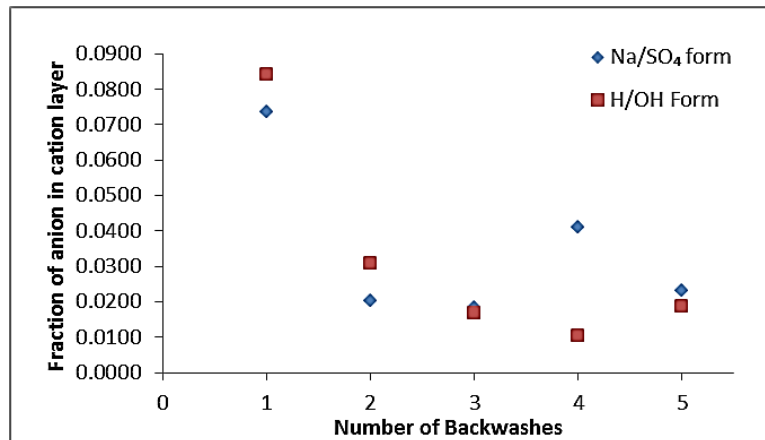


Figure 31. Comparison of anionic fraction at 13.5 gpm and 75% expansion

Results comparing both forms of resin at 13.5 gpm and 150% bed expansion are shown in Figures 32 and 33. Results for anion layer indicate that after five backwashes, cationic fraction in anion layer for Na/SO₄ form resin has a slightly higher value of 0.097% compared to H/OH form (0.063%). Results for cation layer show that lower anionic fraction of 0.014% is observed in Na/SO₄ form after five backwashes compared to 0.023% for H/OH form. This could be due to the increased density of cation resin in Na/SO₄ form resulting in decreased mixing in the cation layer at high bed expansion.

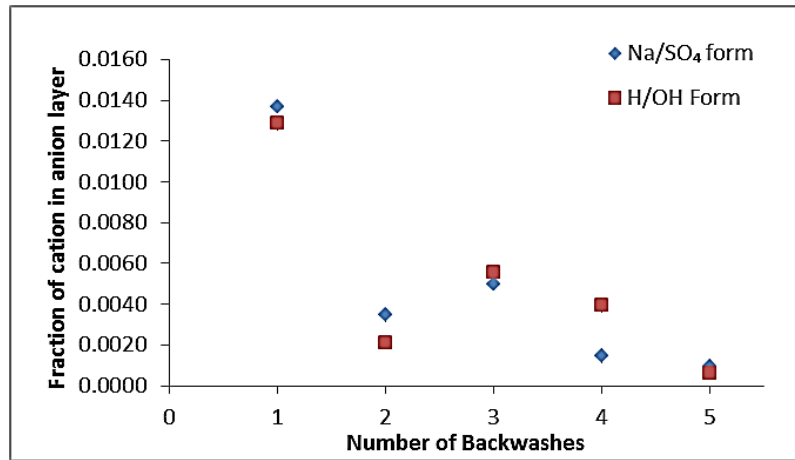


Figure 32. Comparison of cationic fraction at 13.5 gpm and 150% expansion

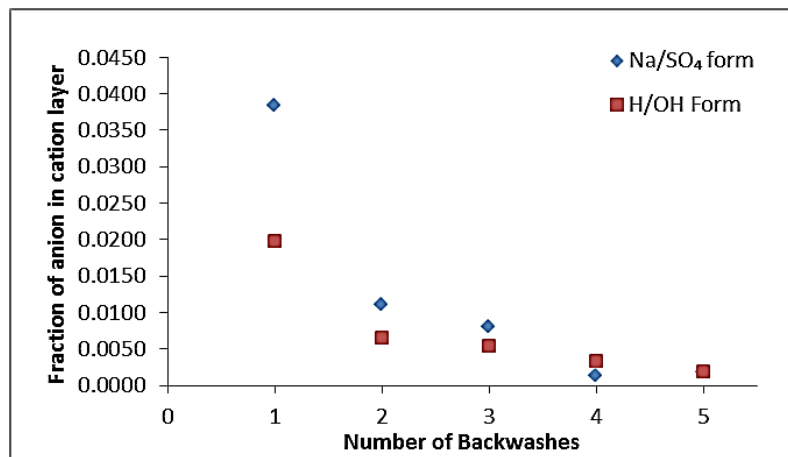


Figure 33. Comparison of anionic fraction at 13.5 gpm and 150% expansion

Results comparing both forms of resin at a low flow rate of 4 gpm and 50% bed expansion are shown in Figures 34 and 35. The results for anion layer indicates fluctuation with each backwash for H/OH form whereas a slightly uniform decrease in cross contamination is noted in the Na/SO₄ form. The final value of cationic fraction is lower for Na/SO₄ form (0.08%) compared to H/OH form (0.2%). Cation layer results show that Na/SO₄ form resin has a lower anionic fraction of 0.13% after five backwashes compared to H/OH form which has 0.23%.

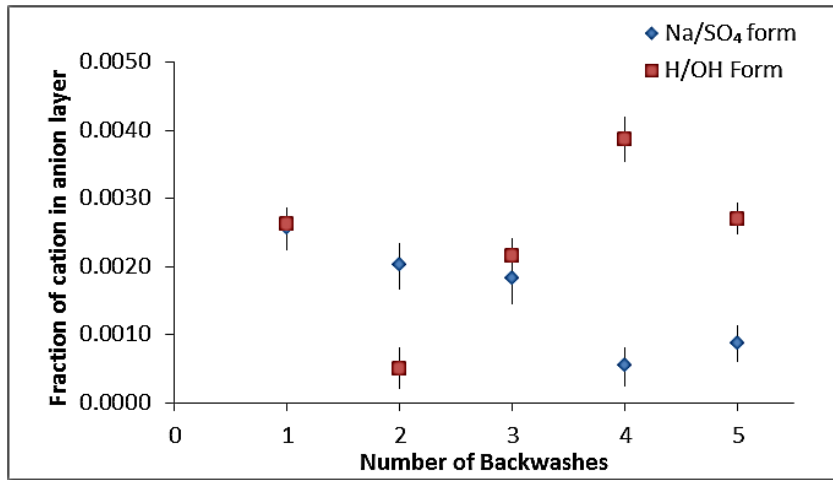


Figure 34. Comparison of cationic fraction at 4 gpm and 50% expansion

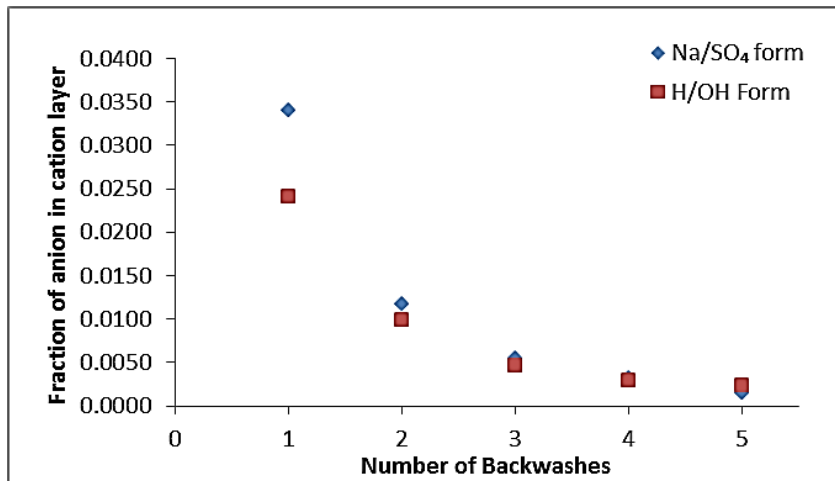


Figure 35. Comparison of anionic fraction at 4 gpm and 50% expansion

Figures 36 and 37 show results comparing both forms of resin at a flow rate of 6.5 gpm and 75% bed expansion. The anion layer has higher cationic fraction of 0.5% in Na/SO₄ form; 12 times greater than that in H/OH form after five backwashes. Results for cation layer show close values for both forms of resin with 0.23% of anionic fraction for Na/SO₄ form and 0.26% for H-OH form.

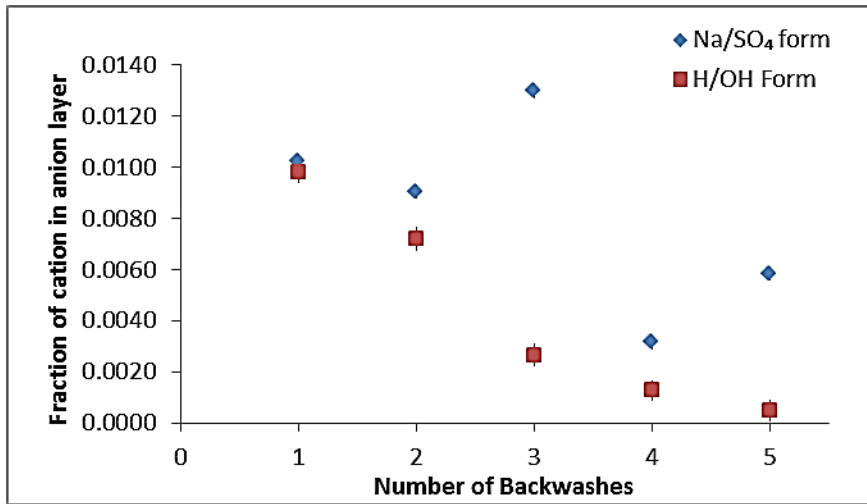


Figure 36. Comparison of cationic fraction at 6.5 gpm and 75% expansion

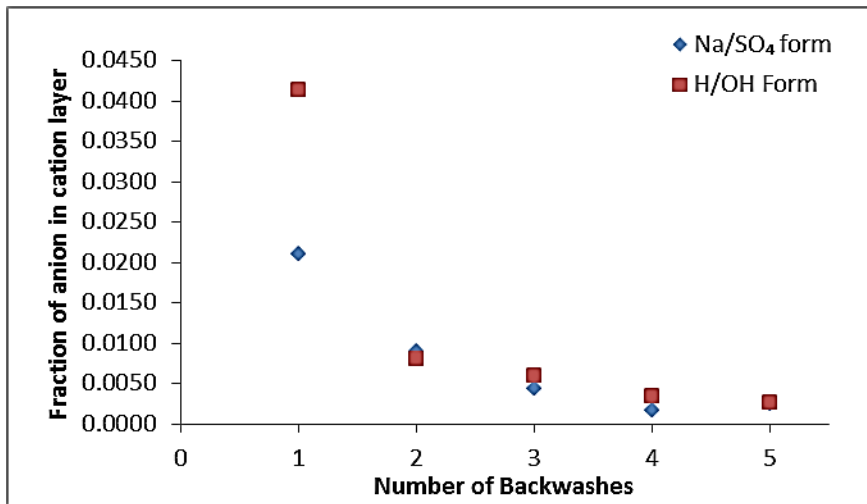


Figure 37. Comparison of anionic fraction at 6.5 gpm and 75% expansion

Results comparing both forms of resin at a flow rate of 6.5 gpm and 100% bed expansion are shown in Figures 38 and 39. The anion layer shows cationic fractions greater 1% for all five backwashes for Na/SO₄ form. The increased bed expansion does not compensate for the mixing effect between the interface and anion layer. The results for cation layer, indicates that the anionic fraction is lower for Na/SO₄ form (0.07%) compared to H/OH form (0.1%) effectively removing the anion resin from the cation layer.

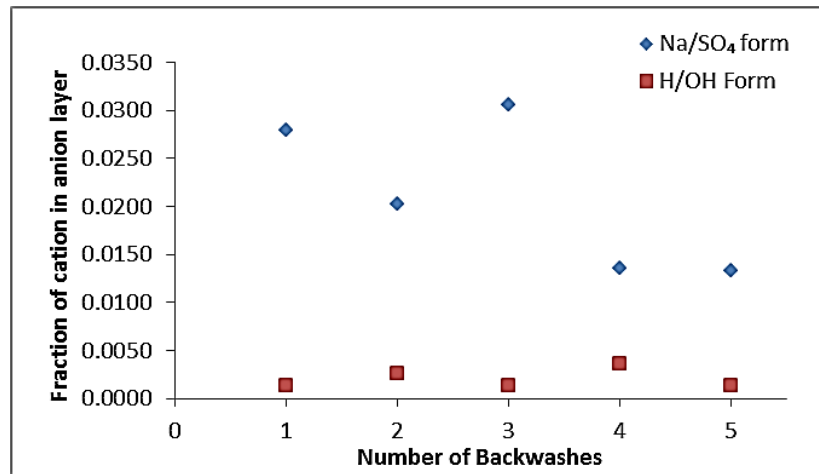


Figure 38. Comparison of cationic fraction 6.5 gpm and 100% expansion

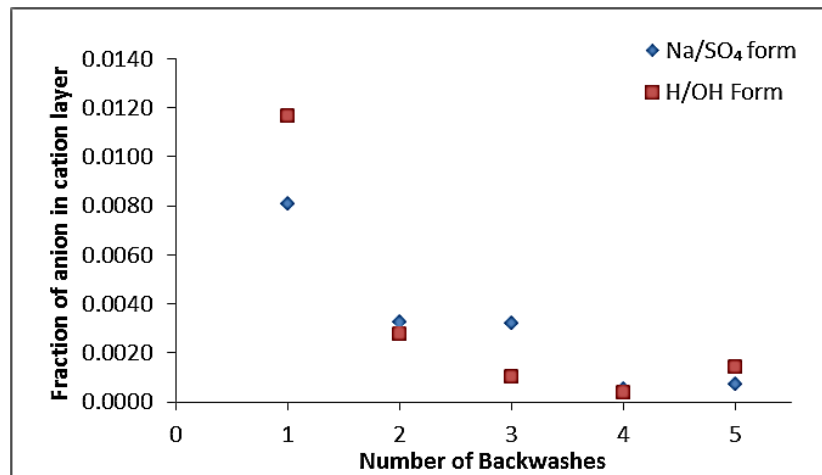


Figure 39. Comparison of anionic fraction at 6.5 gpm and 100% expansion

RESIN TRANSFER STUDY

After separation of resins, efficient removal of anion layer to the anion regeneration vessel and interface layer to the resin storage vessel had to be confirmed. The objective was to verify if interface isolation method was to effectively maintain the purity of both layers during resin transfer.

An initial test was done to fine tune the transfer process. After the gravity separation of cation and anion layers, the CR vessel was completely filled with water. The take-off port near the anion layer was connected to the anion regeneration column through a transfer tube. The column was pressurized to 15 psig before transfer. No visible cross contamination was noted in the anion layer during transfer. After transferring the anion layer, the take-off port near the interface layer was connected to the resin storage vessel. A higher pressure of 20 psig was chosen for interface resin transfer since it had to travel further and line pressure drop was anticipated to be greater. A low degree of mixing was noticed in the interface and cation layer during resin transfer. In both cases, using a low backflow through the cationic bed during transfer helped stabilize the bed. In the interface column there was about 6 inches of cation resin with a total bed height of 8 inches. These heights are primarily a reflection of the location of the interface levels between the sampling/transfer points in the service column.

Two experiments were performed to quantify the effectiveness of the transfer process and determine better conditions for transfer of both resins. The resin bed was initially air mixed for 10 minutes. Gravity separation of the bed was done at 13.5 gpm and 100% bed expansion. The anionic fraction in cation layer was measured after five backwashes. The

volumes of anion and heel resin transferred were measured. Anionic fraction in cation layer was also measured after transfer to ascertain the change in cross contamination during transfer of heel resin. For the first experiment, anion layer was transferred at 20 psi and interface at 30 psig and for the second experiment, anion layer was transferred at 30 psig and interface at 40 psig. After each experiment, both layers were mixed in the resin storage vessel and transferred back to the cation regeneration vessel.

The results for both experiments are compared in Table 2. The purity of the cation layer is not affected by the transfer process. Slightly lower values of anionic fraction is observed due to drain and refilling of column after anion transfer during when more anion resin is pushed towards the interface. In both experiments the pressures were sufficient for effective resin transfer, however, increased pressure (experiment 2) gives the operator better control of the transfer process. Hence there exists a large amount of operator variability and relatively loose correlation between pressure and purity.

Table 2. Resin transfer study

	Experiment 1 (20 & 30 psig)	Experiment 2 (30 & 40 psig)
Volume % of Anion layer transferred =	92.3	91.1
Volume % cation resin left behind =	79.2	77.9
Volume % comprised by heel resin =	16.2	17.2
Anionic fraction in cation layer before transfer =	0.015	0.019
Anionic fraction in cation layer after transfer =	0.014	0.010

DISCUSSION

The flowrates used were compared with the backwash expansion data given by Dowex for both the resins as shown in Figure 40 (DOWEX 650C and 550A MSDS). The lines in the graph indicate the percentage expansion that can be achieved for a set flowrate. This helps to determine the flowrate needed to rinse a homogenous bed to remove fines without losing any resin from the top of the column.

A summary of all the backwash conditions used are shown in Table 3. Though it was easy to operate at the Dow cationic recommended curve, the bed could not be fluidized at the Dow anionic conditions because of the presence, and higher density, of the cationic resin in the mixed bed.

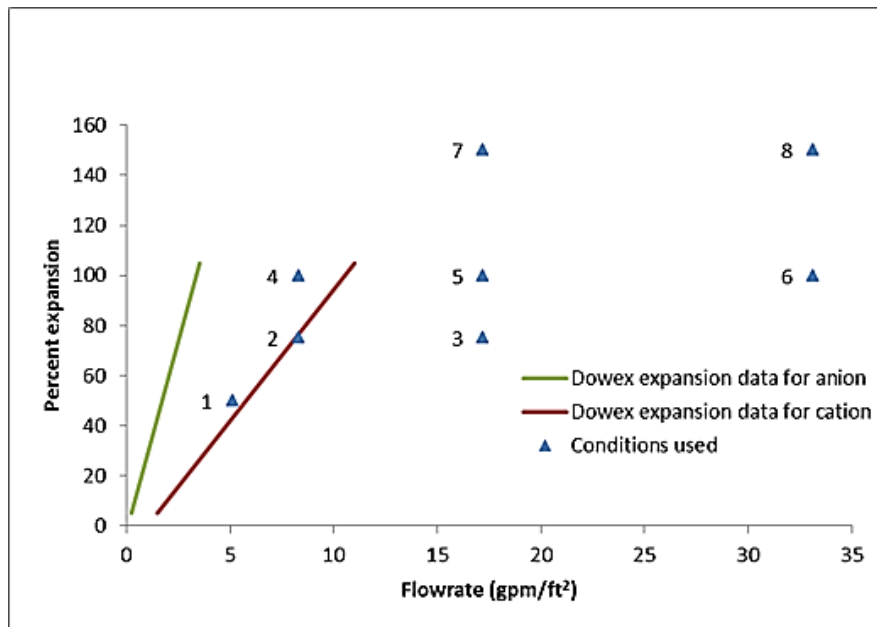


Figure 40. Comparison of conditions used and DOWEX expansion data

Table 3. Summary of backwash conditions used

S no.	Flowrate (gpm)	Flowrate (gpm/ft²)	Percent expansion
1	4.0	5.1	50
2	6.5	8.3	75
3	13.5	17.2	75
4	6.5	8.3	100
5	13.5	17.2	100
6	27.0	33.1	100
7	13.5	17.2	150
8	27.0	33.1	150

The effect of flow rate on mixing of resin beads during backwash could be visually noticed and without sufficient expansion, results in increased cross contamination. Reynolds numbers of the flow rates used can indicate the degree of viscous effects experienced by the resin beads.

Table 4 shows the column Reynolds numbers corresponding to the flow rates used for the backwash experiments. 4 and 6.5 gpm flows lie within the laminar region whereas flow rates of 13.5 gpm and 27 gpm lie in the transition and turbulent regions respectively. High Reynolds numbers indicate that the viscous effects acting on the resin beads are greater. This causes increased mixing as the bed expands. No analysis of particle Reynolds numbers in the bed were made.

Table 4. Estimation of column Reynolds numbers from flow rates

Flow rate (gpm)	Velocity (m/s)	Reynolds number
4	0.0035	1050
6.5	0.0056	1710
13.5	0.0117	3538
27	0.0233	7061

Out of the 64 samples from the second backwash or later in all experiments for H/OH form resin, only one data point exceeded 1% cross contamination. For the first backwashes, only once did the cationic resin in anion layer exceed 1%. The lowest flow rate required to achieve 100% expansion was found to be 6.5 gpm and 13.5 gpm was the minimum flowrate required to achieve 150% expansion. Na/SO₄ form resin backwash results indicate that increased backwash time is required for better separation.

A working model can be developed to predict the separation and redistribution of the resin beads with each backwash. The first step towards realization of such a model would be to develop equations to predict the bead rise and fall characteristics. While developing a force balance around a single resin bead, the forces to be taken into consideration are the drag force F_D , force due to buoyancy F_B , force acting on an accelerating body F_A and gravitational force due to the weight W of the bead. The directions of forces acting on a spherical bead during rise and fall are shown in figure 41.

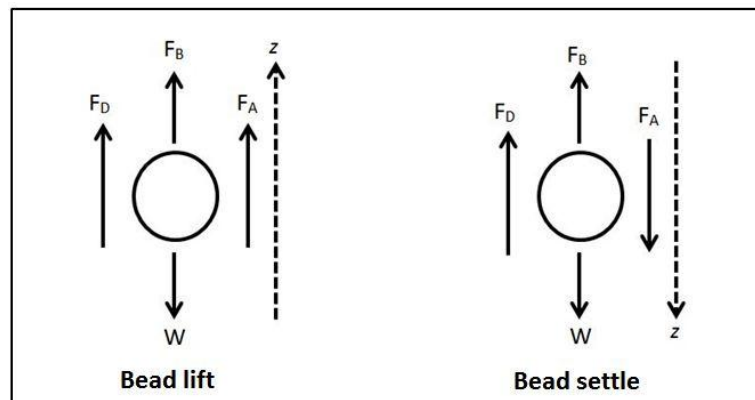


Figure 41. Force balances for rise and fall of a resin bead

Equations (1) and (2) show the change in velocity over time for rise and fall of a single resin bead. The derivations for both equations are presented in Appendix C.

Bead rise:

$$\frac{dv}{dt} = \frac{1}{A}(B - C * v * |v| * C_d) \quad (1)$$

Where,

$$A = 1 + \frac{1}{2}\bar{\rho}, B = (1 - \bar{\rho})g, C = \frac{3\bar{\rho}}{4d}, \bar{\rho} = \frac{\rho_f}{\rho}$$

Bead fall:

$$\frac{dv}{dt} = \frac{1}{A}(B + C * (U - v) * |(U - v)| * C_d) \quad (2)$$

Where,

$$A = 1 + \frac{1}{2}\bar{\rho}, B = (\bar{\rho} - 1)g, C = \frac{3\bar{\rho}}{4d}, \bar{\rho} = \frac{\rho_f}{\rho}$$

CONCLUSION AND RECOMMENDATIONS

The following conclusions are drawn from this study:

- Higher flowrate causes increased mixing due to turbulence whereas lower flowrate gives significantly less cross contamination.
- Greater bed expansion gives better separation at the same flow rate.
- Increased water temperature leads to better separation of anion resin from cation layer and decrease in cross contamination in anion layer with each backwash.
- Interface isolation method is a viable resin transfer technique at pressures of 30/40 psig enabling better control during operation. However, loose correlation is observed between pressure and purity since there is a large amount of operator variability.

Possible future work includes defining different flow and expansion conditions for each of the five backwashes to achieve low cross contamination. It is possible that once the bed has a first backwash and a primary separation, that the next backwash could be performed at a somewhat lower flowrate. The top half of the bed would still expand while providing less turbulence and mixing at the interface. If the primary concern is to liberate more cationic beads from the anionic resin this method may have some advantage. While if the concern is the release of anionic beads from the cationic resin then there must be sufficient flow to expand the cationic resin regardless.

The impact of steady state expansion from a high to low flow rate in a single backwash also needs to be addressed. The water property with the largest variation over the range of

potential temperatures is viscosity. The impact of this factor can be evaluated in future studies for different backwash conditions.

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APPENDIX A

RESIN SEPARATION PILOT PLANT – OPERATING MANUAL

INTRODUCTION

The purpose of this operating manual is to provide all necessary information for the user to effectively conduct resin separation studies on the pilot plant. Resin separation is the most important step in the resin regeneration process. In mixed bed ion exchange, cation and anion resin are present side by side, thoroughly mixed. Ineffective separation prior to regeneration results in fully loaded cross contaminated beads with counterions and subsequently increasing leakage of ions into the water. The topics covered are as follows,

- Resin separation efficiency by gravity separation technique
- Cross contamination measurement after separation
- Resin transfer by interface separation method

EQUIPMENT SETUP

The assembled pilot plant (Figure 28) is comprised of the following units:

- **DILUTION/ DEMIN WATER RESIN SLUICE PUMP**

CAPACITY: 4 m³/ h

DISCHARGE PRESSURE: 50 PSIG

MOTOR: 3 phase

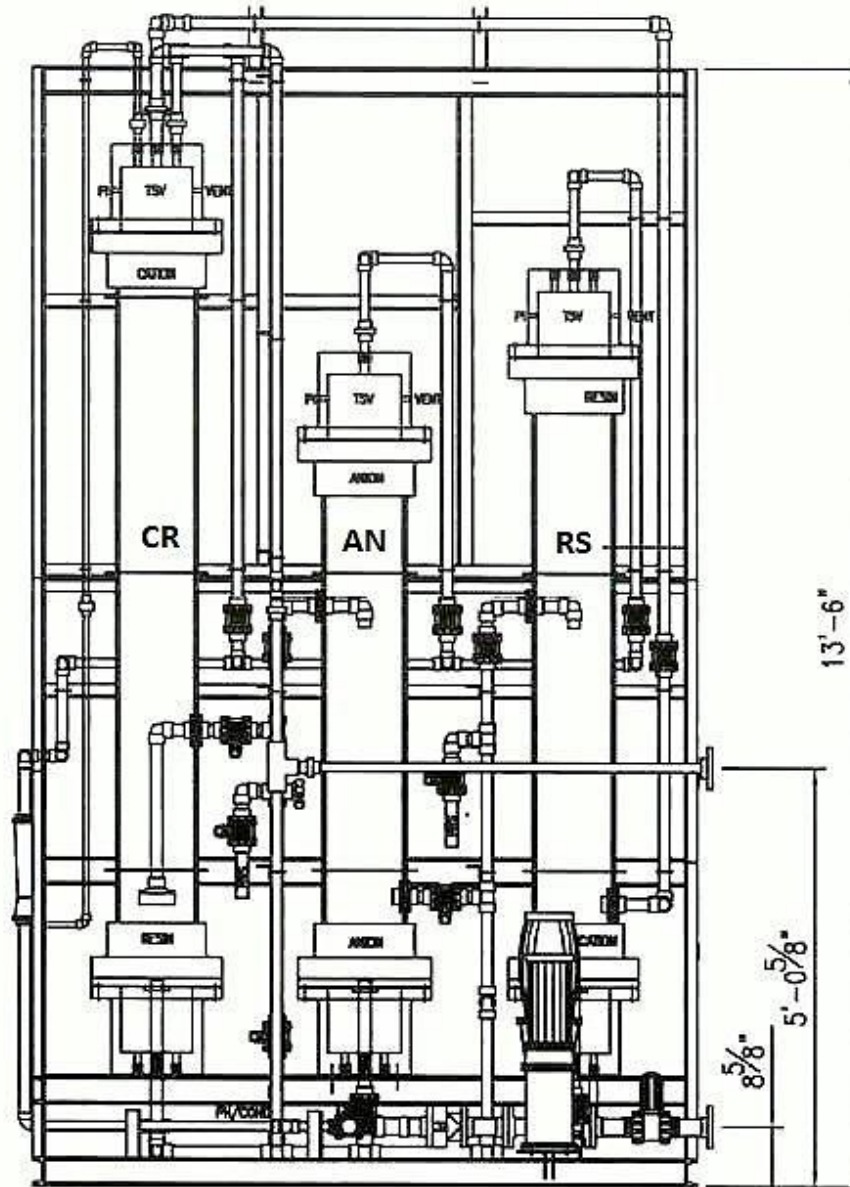


Figure 1. Resin separation Pilot plant (Aquatech International Corp.)

- **CATION REGENERATION/SEPARATION VESSEL (CR)**

DIAMETER: 304.8 mm

DESIGN PRESSURE: 75 PSIG

MATERIAL: PLASTIC or FRP or CS Epoxy Lined

STRAIGHT HEIGHT: 1500 mm

- **ANION REGENERATION VESSEL (AR)**

DIAMETER: 304.8 mm

DESIGN PRESSURE: 75 PSIG

MATERIAL: PLASTIC or FRP or CS Epoxy Lined

STRAIGHT HEIGHT: 1724 mm

- **RESIN STORAGE VESSEL (RS)**

DIAMETER: 304.8 mm

DESIGN PRESSURE: 75 PSIG

MATERIAL: PLASTIC or FRP or CS Epoxy Lined

STRAIGHT HEIGHT: 1724 mm

- **DI WATER STORAGE TANK**

MATERIAL: PPL or equivalent

- **RESIN USED**

CATION RESIN: DOWEX 650C(H)

ANION RESIN: DOWEX 550A(OH)

SAFETY AND ENVIRONMENTAL ISSUES

Water should be kept away from the computers and electrical equipment to avoid electric shock and electrocution. Any spilled water should be immediately dried to avoid slipping.

Use caution to prevent tripping in the work area. Use caution while climbing the ladder to reach the top of the tanks. Always be aware of the surroundings and wear safety glasses at all times. The water used for this testing is mostly recycled and the pure water obtained may be used for other experiments. The resins are not hazardous to health. Resin spilled

on the floor however is a slip hazard. Avoid skin contact with sodium hydroxide, sulphuric acid and ammonia (Material Safety Data Sheets).

Utmost care is required while handling the valves and piping since increased physical strain can lead to breakage. In case the resins are regenerated, the sodium hydroxide and sulphuric acid used as regenerants are mixed in a separate tank after completion of the process to bring pH back to acceptable levels before disposal.

TEST PROCEDURE – OVERVIEW

The three vessels in the pilot plant as shown in Figure 1 are:

- Cation Regeneration (CR) vessel
- Anion Regeneration (AN) vessel
- Resin Storage (RS) vessel

Cation resin and anion resins are separated in the Cation Regeneration / Separation vessel (CR) by backwashing and by gravity, cation resin being heavier will settle below the anion resin component. An air scrub/scour is used to mix resins and create a homogenous bed prior to separation. Gravity separation of the two resins is performed using step-by-step backwash runs. This is performed by fixing a backwash flow rate and resin bed expansion height (e.g. 100% resin expansion, 75% resin expansion etc.) To determine a specific resin expansion, refer to resin manufacturer's specification (resin expansion rate vs. backwash flow linear velocity) and the required backwash flow rate will be determined considering the cross sectional area of the test separation vessel.

During backwashing steps the DI water enters from the bottom of the vessel containing resins in an upward direction thereby fluidizing the resin bed. Although there is a sharp

difference between in resin particle densities and particle size distribution, even after gravity separation there is a level of intermixing at the interface. Separation of the resin is directly proportional to degree of fluidization of the mixed resin. Degree of fluidization is affected by the backwash velocity of water and duration of backwash. The anion layer on top is transferred to the Anion Regeneration (AN) vessel and the interfacial layer is transferred to the Resin Storage (RS) vessel.

PRE-OPERATION CHECKS

Before operating the pilot plant, ensure that:

- Water level in the DI tank is adequate
- Instruments in the pilot plant are in good working condition
- All leaks are fixed
- Availability of air is checked

WATER FILLING AND HYDRO TESTING

The filling and hydro testing process is done one tank at a time. Check that all valves are closed before beginning the test.

CR VESSEL:

- Open the valve **H1** (Lab's RO water inlet) to fill the DI water tank if the tank is not filled. Ensure the tank is filled no closer than a foot from the top.
- Open valve **DI1** and **DI4**. Check for leaks around the pump.
- Partially open ($\frac{1}{4}$ turn) the recycle valve **DI6**. Refer figure 29 for valve locations.
- Now open valves **CR1** (inlet valve to CR tank) and the vent valve **CR2** on the top.

- Turn on the **DI** pump. Check **PID-1** and flow indicator **FL2**. Ensure pressure is not more than 55 psig while the CR tank is getting filled.
- Now close valve **DI6** completely.
- During the fill check for any leaks.
- Fill CR vessel until water drains out from the vent and then close valve **CR2**.
- Let the pressure rise up to 55 psig. Turn off the pump and look for pressure decay. Wait for ten minutes. Pressure drop of about 2-3psi is admissible.
- Now close valve **CR1**.

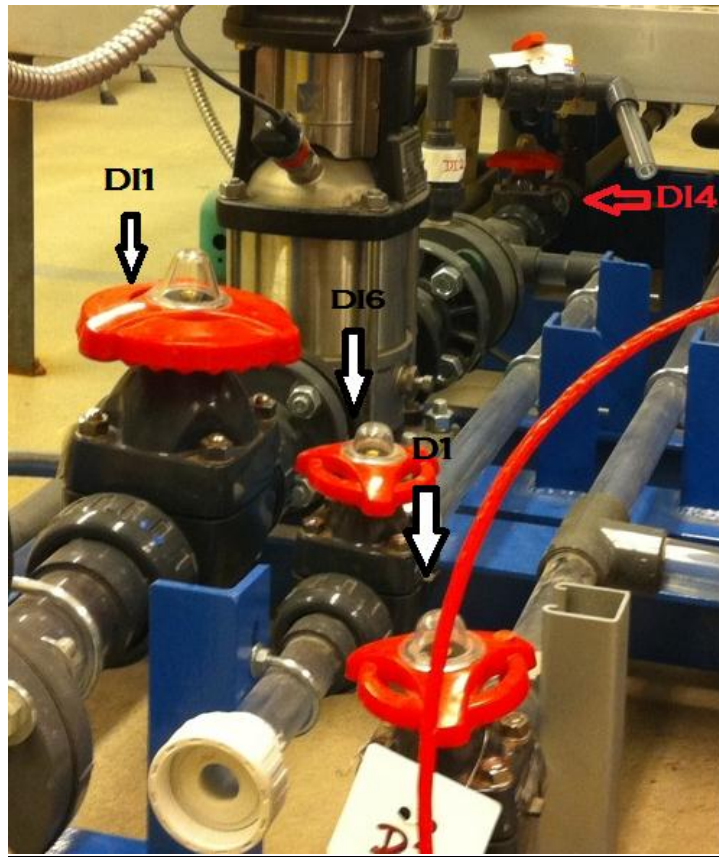


Figure 2. Inlet and recycle valves

AN VESSEL:

- Keep valve **DI6** partially open (¼ turn).
- Open inlet valve **AN1** and vent valve **AN2**.
- Turn on the **DI** pump and set pump speed to level 3. Check **PID-1** and flow indicator **FL2**. Ensure pressure is not more than 55 psig while the AN vessel is getting filled.
- Now close valve **DI6** completely.
- Check for leaks during the filling procedure.
- Fill the vessel until water drains out from the vent and close vent valve **AN2**.
- Let pressure increase to 55 psig then turn off the pump and look for pressure decay.
Wait for ten minutes. Pressure drop of about 2-3psi is admissible.
- Now close the valve **AN1**.

RS VESSEL:

- Keep valve **DI6** partially open (¼ turn).
- Open valve **RS1** and vent valve **RS2**.
- Turn on the **DI** pump and set pump speed to level 3. Check **PID-1** and flow indicator **FL2**. Ensure pressure is not more than 55 psig while the AN vessel is getting filled.
- Now close valve **DI6** completely.
- Check for leaks during the filling procedure.
- Fill the tank until water drains out from the vent and close the vent valve **RS2**.
- Let pressure increase to 55 psig then turn off the pump and look for pressure decay.
Wait for ten minutes. Pressure drop of about 2-3psi is admissible.
- Now close the valve **RS1**.

RECYCLING WATER TO DI TANK FROM VESSELS

In all three vessels water can be drained back to the DI tank by gravity and by pressurising vessels with air. Ensure the pressure gage for air supply inlet is at 10 psig and the air inlet valve **A1** is open.

CR VESSEL:

- Open CR vessel bottom valve **CR14** and drain valve **D1**.
- Close vent valve **CR2**.
- Open air valve **A2** to begin building pressure within CR vessel which will force water to flow back in to the DI tank.
- Close and open air valve **A2** occasionally to regulate water flow.
- Once all the water has been recycled, close valve **CR14** and then open **D1**.
- Open vent valve **CR2** slowly to remove air from the vessel and decrease pressure.

AN & CR VESSELS:

- Open drain valve **D1**. Let AN vessel bottom valve, **AN13** and vent valve **AN2** be closed.
- Open air valve **AN10** in the bottom to start bubbling air into AN vessel until pressure in the vessel is the same as the inlet air pressure.
- Now open bottom valve **AN13** to let the excess air push the water back into the DI tank.
- Repeat the above steps till all water is recycled from AN vessel.
- After the recycling is complete open vent valve **AN2**.

- The process is similar for RS vessel where **RS2** is the vent valve, **RS12** is the drain valve and **RS8** is the air inlet valve.

RESIN LOADING

A specific amount of cation and anion resin can be loaded into the Resin Separation vessel. The cation resin is filled first followed by the anion resin. Make sure the DI tank is full before beginning this procedure.

- Open bottom valve **CR14** and drain valve **D1**. Close vent valve **CR2** and open Air inlet **A2** to facilitate effective recycling of water back into the DI tank.
- Once water level comes down to about a foot from the bottom of the vessel, close valve **A2** and slowly open vent valve **CR2** to remove excess air from the CR vessel.
- Connect the transfer hose leading from the cation drum to valve **E1** which in turn leads to the resin eductor. Ensure **E1** is closed initially.
- Open valve **R1** (present beneath the DI tank). Close the bottom valve **CR14** and drain valve **D1**.
- Keep valve **DI6** partially open (¼ turn). Turn on the pump and increase to maximum speed.
- Check **FI1** to note flow and then close valve **DI6**.
- Open valve **E1** to create suction. Now insert the transfer hose into the resin drum while stirring the resin gently.
- Turn off the pump once desired amount is transferred. Wait for excess resin to return back to the resin drum and close valve **E1**.
- By visual observation, allow time for the resin to settle.

- If desired amount of resin was not transferred, drain the CR vessel and repeat the process.
- Connect Valve **E1** to anion drum and repeat the process to transfer required amount of anion resin to the CR vessel.

RESIN SEPARATION

The cation and anion resin beads are separated by virtue of difference in their densities. This separation is achieved during the backwash and settling procedure where the less dense anion resin forms a top layer and the slightly heavier cation resin forms the bottom layer. Before separation the resin bed has to be air mixed to form a homogenous mixture.

AIR MIXING:

Ensure that the water level is only an inch above the resin bed prior to air mixing. Open the air inlet valve **CR11** and vent valve **CR2**.

- Open the air inlet valve **A1** and maintain a flow rate of at least 4 m³/hr.
- Carryout the air mixing process for 10 minutes.
- Close valves **A1** and **CR11**.
- Allow two minutes for the resin mixture to settle.

BACKWASH & SETTLING STEPS:

- Open valves **CR14** and vent valve **CR2**. Let valve **DI6** be partially open and ensure all other valves are closed.
- Turn on the pump while set at minimum speed and check **FI1** to note flow.
- Close valve **DI6** and adjust pump speed to obtain desired flowrate.

- The anion and cation resins separate during the back wash.
- Continue backwash till bed reaches the desired level of expansion.
- Stop the pump. Close bottom valve **CR14** and allow time for resin to settle.
- The anion resin should now be on top and cation resin in the bottom.
- The interface consists of a mixture of both anion and cation resins. It is called the 'heel resin'.
- Drain water back as shown in section 4.4 and repeat backwash if necessary.

MEASURING CROSS CONTAMINATION

Once resin separation is completed efficiency of separation can be measured by drawing samples from the take-off ports (figure 30).

- Recycle all water from the CR vessel to the DI tank.
- Open the chosen take-off port and remove the port insert.
- Insert a glass tube through the port to remove approximately 5-10 grams of resin.
- Transfer sample to a tall glass vial containing 20% NaCl solution. Allow resins to settle.
- The NaCl will allow the resins to separate due to difference in density.
- Decant or siphon the less dense anion beads from the top layer of the solution and place on filter paper. Allow excess solution to drain from the beads.
- Remove the cation resin beads from the remaining mixture using a sieve or filter and let dry.
- Wash both sets of resin beads with water to dissolve any salt residue and oven dry the samples overnight at 35°C.

- Once dry, weigh the filter paper containing the resin. Remove resin from the filter paper and weigh filter paper separately.
- The weight of each resin can now be determined by subtracting the weight of the filter paper from the weight of the resin and filter paper combination.
- Record weights of anion and cation resins and calculate relative weight fractions.

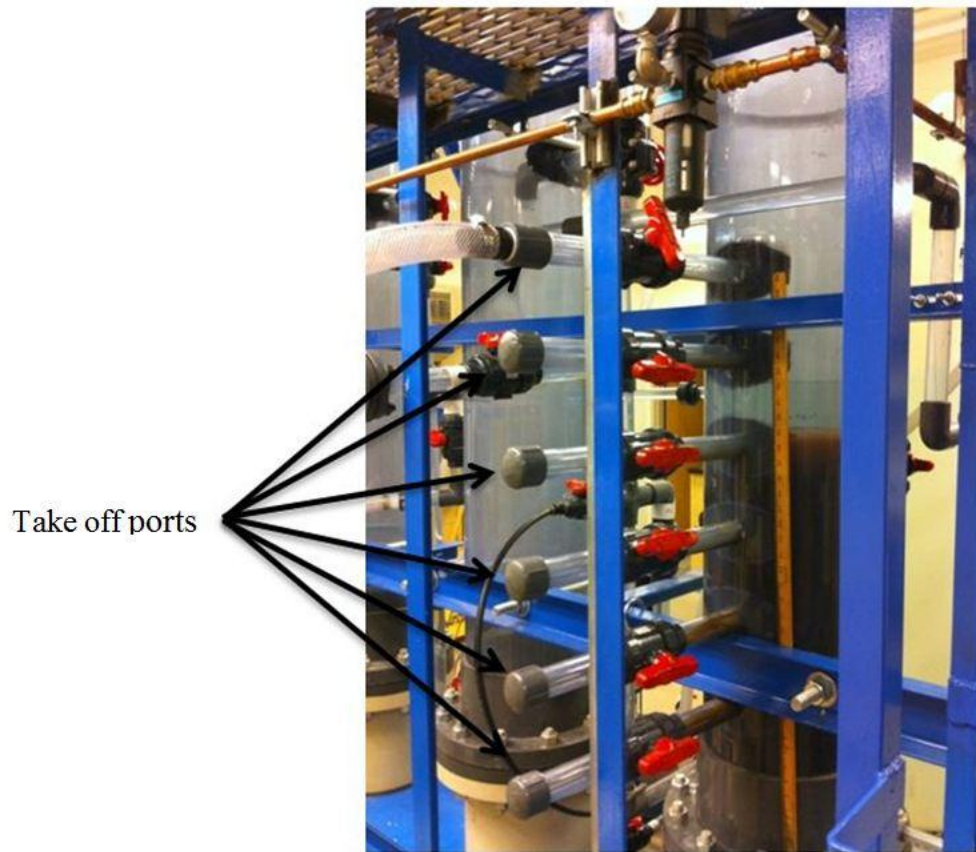


Figure 3. Resin transfer and sampling take-off ports

RESIN TRANSFER

In this step the anion resin is transferred to the AN vessel and the heel resin is transferred to the RS vessel through the take-off ports as shown in Figure 30. Ensure all valves are closed before beginning the procedure.

ANION RESIN TRANSFER TO AN VESSEL:

- Connect the chosen take-off port to **AN5** using the resin transfer tube. Ensure both valves are closed.
- Open the vent valves **AN2** and **CR2**.
- Open the valve **DI6** partially ($\frac{1}{4}$ turn) and turn on the pump while set at minimum speed.
- Note the flow **FI1** and open valve **CR1** to start filling the CR vessel. Close valve **DI6**.
- Once water flows out of the vent, close **CR2** and allow CR vessel to pressurize up to 25 psig.
- To begin resin transfer open the selected take-off valve and then open valves **AN5** and **AN8**.
- Open the bottom valve **CR14** to stabilize the bed during transfer.
- Once the transfer is complete open valve **DI6**. Close valves **AN5** and **AN8**.
- Turn off the pump and close valves **CR1**, **CR14** and **DI6**.
- Wash and collect the resin remaining in the resin transfer tube separately.

HEEL RESIN TRANSFER TO RS VESSEL:

Select an appropriate take-off port from CR vessel (**CR5** to **CR10**) for transferring the heel resin to RS vessel.

1. Connect the resin transfer tube from chosen port to **RS13**. Ensure both valves are closed.
2. Open vent valves **CR2** and **RS2**.
3. Open valve **DI6** partially ($\frac{1}{4}$ turn) and turn on the pump while set at minimum speed.

4. Note the flow **FI1**. Open valve **CR1** and close valve **DI6**.
5. Once water flows out of the vent close **CR2** and allow CR vessel to pressurize up to 25 psig.
6. Open selected take-off valve, and **RS13** leading to the RS vessel.
7. Open bottom valve **CR14** to stabilize bed during transfer.
8. After Transfer is complete open valve **DI6**. Close the take-off valve and **RS13**.
9. Close **DI4**, **CR1** and **CR14** to stop supply of water to CR vessel.
10. Turn off the pump and close the valve **DI6**.

RESIN TRANSFER TO CR VESSEL FROM AN AND RS VESSELS:

Transfer of resin to CR vessel from AN and RS vessel is done in two steps. The anion resin in the AN vessel is first transferred to the RS vessel. The resin from the RS vessel is then transferred to the CR vessel.

1) Transfer from AN vessel to RS vessel

- Open valves **AN1**, vent valves **AN2** and **RS2**. Keep valve **DI6** partially open (¼ turn).
- Start pump to begin filling the AN vessel with water at a flowrate of 8 gpm.
- Close valve **DI6** and close off vent valve **AN2** as soon as water flows out.
- Let the pressure in AN vessel increase up to 25 psig.
- Now open **AN9** and **RS6** to begin transfer. Open bottom valve **AN13** to lift the bed while transfer takes place.
- The transfer process cannot be completed in one shot. Once the RS vessel fills up, close **RS6** and **AN9**. Open **DI6** and turn the pump off.

- Drain water from RS vessel and repeat the transfer process till most of the resin has been transferred.
- The resin remaining in the bottom can be transferred through the bottom resin transfer pipe.
- Pressurize AN vessel again up to 25 psig. Open valves **AN14** and **RS6** to transfer the remaining resin.
- Repeat above step to ensure complete transfer.
- Drain water from AN vessel to the DI tank.

2) *Transfer from RS vessel to CR vessel*

- Open valves **RS1**, vent valves **RS2** and **CR2**. Keep valve **DI6** partially open (¼ turn).
- Start pump and begin filling RS vessel water at a normal flowrate.
- Close valve **DI6** and vent valve **RS2** as soon as water flows out.
- Let the pressure in RS vessel increase up to 25 psig.
- Open valve **RS7** to start transfer and open bottom valve **RS12** to slowly lift the bed while transfer takes place.
- When the CR vessel reaches maximum water capacity, drain water from the CR vessel and repeat steps 4 and 5 until 50% of the resin is transferred.
- To transfer the bottom portion of the resin the resin can be transferred through the bottom valve **RS9**.
- Connect the resin transfer hose from the topmost take-off port to **AN5**.
- Drain water completely from the CR vessel before beginning the transfer.
- Pressurize RS vessel to 25 psig and open valves **RS9**, **AN5** and the take-off port valve to transfer the resin.

- Drain CR vessel and repeat above step to ensure complete transfer of resin.
- Recycle excess water back to the feed tank and close off all valves after the transfer is complete.

RESIN REGENERATION

- After gravity separation, transfer the anion resin to the AN vessel and the heel resin to the RS vessel.
- Based on the total gram equivalents of anion and cation resin present in the AN and CR vessels respectively, caustic and sulphuric acid solutions containing the same gram equivalents of OH^- and H^+ ions have to be prepared.
- Transfer the prepared solutions in to the columns using a chemical pump. For CR vessel, the top most take-off port can be used as an inlet for the sulphuric acid solution. For AN vessel, there exists an auxiliary inlet at the top of the vessel for transferring the caustic solution.
- Let the resins soak for 45 minutes after which the sulphuric and caustic solutions should be drained safely to the storage tank in the basement. This followed by rinsing the resins with DI water which is also sent to the storage tank.
- Fill the CR vessel completely with water and slowly rinse the bed while measuring the conductivity of the exiting the water stream using the conductivity sensor. After the conductivity reaches a sufficiently low value, the water can be recycled back to the DI tank during the rinse process. The same process is repeated for the AN vessel.
- Ensure that the solution in storage tank is neutralised before it is dumped.

APPENDIX B
EXPERIMENTAL DATA

INTRODUCTION

The following experimental data shows change in position of the interface with each backwash for different experimental conditions. Interfacial height gives a visual perception of how well the anion and cation resin separate after each backwash. The tables also show the anion and cation sampling distance relative to the interface. Other details mentioned are the air mix conditions and the total bed height.

H/OH FORM

Experiment 1: 13.5 gpm and 100% bed expansion

Air mixing		Bed Conditions		
Rate (m ³ /h)	Time (min)	Cation sampling height (cm)	Anion sampling height (cm)	Bed height (cm)
3.5	10	25	54	77
Runs	Interface height (cm)	Sampling distance from interface (cm)		
		Cation resin	anion resin	
1	59.0	18.0	13.0	
2	52.0	11.0	20.0	
3	50.0	9.0	22.0	
4	49.5	8.5	22.5	
5	49.4	8.4	22.6	

Experiment 2: 13.5 gpm and 150% bed expansion

Air mixing		Bed Conditions		
Rate (m ³ /h)	Time (min)	Cation sampling height (cm)	Anion sampling height (cm)	Bed height (cm)
3.5	10	25	54	76
Runs	Interface height (cm)	Sampling distance from interface (cm)		
		Cation resin	anion resin	
1	49.0	8.0	23.0	
2	47.0	6.0	25.0	
3	46.0	5.0	26.0	
4	46.0	5.0	26.0	
5	46.0	5.0	26.0	

Experiment 3: 13.5 gpm and 75% bed expansion

Air mixing		Bed Conditions		
Rate (m ³ /h)	Time (min)	Cation sampling height (cm)	Anion sampling height (cm)	Bed height (cm)
3.5	10	25	54	76
Runs	Interface height (cm)	Sampling distance from interface (cm)		
		Cation resin	anion resin	
1	57.0	16.0	15.0	
2	50.5	9.5	21.5	
3	49.0	8.0	23.0	
4	48.5	7.5	23.5	
5	48.5	7.5	23.5	

Experiment 4: 6.5 gpm and 100% bed expansion

Air mixing		Bed Conditions		
Rate (m ³ /h)	Time (min)	Cation sampling height (cm)	Anion sampling height (cm)	Bed height (cm)
3.5	10	25	54	77
Runs	Interface height (cm)	Sampling distance from interface (cm)		
		Cation resin	anion resin	
1	50.5	9.5	21.5	
2	49.2	8.2	22.8	
3	49.0	8.0	23.0	
4	48.8	7.8	23.2	
5	48.5	7.5	23.5	

Experiment 5: 6.5 gpm and 75% bed expansion

Air mixing		Bed Conditions		
Rate (m ³ /h)	Time (min)	Cation sampling height (cm)	Anion sampling height (cm)	Bed height (cm)
3.5	10	25	54	75
Runs	Interface height (cm)	Sampling distance from interface (cm)		
		Cation resin	anion resin	
1	51.5	10.5	20.5	
2	49.0	8.0	23.0	
3	48.5	7.5	23.5	
4	48.0	7.0	24.0	
5	48.0	7.0	24.0	

Experiment 6: 4 gpm and 50% bed expansion

Air mixing		Bed Conditions		
Rate (m ³ /h)	Time (min)	Cation sampling height (cm)	Anion sampling height (cm)	Bed height (cm)
3.5	10	25	54	76
Runs	Interface height (cm)	Sampling distance from interface (cm)		
		Cation resin	anion resin	
1	49.5	8.5	22.5	
2	47.5	6.5	24.5	
3	47.0	6.0	25.0	
4	46.5	5.5	25.5	
5	46.0	5.0	26.0	

Experiment 7: 6.5 gpm and 75% bed expansion (40°C)

Air mixing		Bed Conditions		
Rate (m ³ /h)	Time (min)	Cation sampling height (cm)	Anion sampling height (cm)	Bed height (cm)
3.5	10	25	54	75
Runs	Interface height (cm)	Sampling distance from interface (cm)		
		Cation resin	anion resin	
1	51.0	10.0	21.0	
2	48.5	7.5	23.5	
3	47.5	6.5	24.5	
4	47.0	6.0	25.0	
5	47.0	6.0	25.0	

Na/SO₄ FORM

Experiment 1: 13.5 gpm and 100% bed expansion

Air mixing		Bed Conditions		
Rate (m ³ /h)	Time (min)	Cation sampling height (cm)	Anion sampling height (cm)	Bed height (cm)
3.5	10	25	54	59.5
Runs	Interface height (cm)	Sampling distance from interface (cm)		
		Cation resin	anion resin	
1	43.5	18.5	10.5	
2	39.5	14.5	14.5	
3	39.5	14.5	14.5	
4	38.5	13.5	15.5	
5	38.0	13	16	

Experiment 2: 13.5 gpm and 75% bed expansion

Air mixing		Bed Conditions		
Rate (m ³ /h)	Time (min)	Cation sampling height (cm)	Anion sampling height (cm)	Bed height (cm)
3.5	10	25	54	59.5
Runs	Interface height (cm)	Sampling distance from interface (cm)		
		Cation resin	anion resin	
1	42.5	17.5	11.5	
2	39.0	14.0	15.0	
3	38.0	13.0	16.0	
4	38.0	13.0	16.0	
5	37.5	12.5	16.5	

Experiment 3: 13.5 gpm and 150% bed expansion

Air mixing		Bed Conditions		
Rate (m ³ /h)	Time (min)	Cation sampling height (cm)	Anion sampling height (cm)	Bed height (cm)
3.5	10	25	54	59.5
Runs	Interface height (cm)	Sampling distance from interface (cm)		
		Cation resin	anion resin	
1	40.5	15.5	13.5	
2	37.5	12.0	16.5	
3	37.0	12.0	17.0	
4	37.0	12.0	17.0	
5	36.5	11.5	17.5	

Experiment 4: 4 gpm and 50% bed expansion

Air mixing		Bed Conditions		
Rate (m ³ /h)	Time (min)	Cation sampling height (cm)	Anion sampling height (cm)	Bed height (cm)
3.5	10	25	54	59
Runs	Interface height (cm)	Sampling distance from interface (cm)		
		Cation resin	anion resin	
1	39.5	14.5	14.5	
2	37.5	12.5	16.5	
3	37.0	12.0	17.0	
4	37.0	12.0	17.0	
5	37.0	12.0	17.0	

Experiment 5: 6.5 gpm and 75% bed expansion

Air mixing		Bed Conditions		
Rate (m ³ /h)	Time (min)	Cation sampling height (cm)	Anion sampling height (cm)	Bed height (cm)
3.5	10	25	54	58
Runs	Interface height (cm)	Sampling distance from interface (cm)		
		Cation resin	anion resin	
1	38.0	13.0	16.0	
2	37.0	12.0	17.0	
3	36.5	11.5	17.5	
4	36.5	11.5	17.5	
5	36.3	11.3	17.5	

Experiment 6: 6.5 gpm and 100% bed expansion

Air mixing		Bed Conditions		
Rate (m ³ /h)	Time (min)	Cation sampling height (cm)	Anion sampling height (cm)	Bed height (cm)
3.5	10	25	54	58
Runs	Interface height (cm)	Sampling distance from interface (cm)		
		Cation resin	anion resin	
1	37.0	12.0	17.0	
2	36.7	11.7	17.3	
3	36.0	11.0	18.0	
4	36.0	11.0	18.0	
5	36.0	11.0	18.0	

APPENDIX C

BEAD RISE AND FALL MODEL

The following forces need to be taken into consideration to model the rise and fall of a spherical resin bead in water:

1. The gravitational pull, $W = mg$
2. The buoyant force F_B , which is equal to the weight of the weight of the fluid displaced by the body. It has the expression $F_B = m_f g$
3. The force on an accelerating body, F_A , which is the kinetic energy associated with moving the resin bead against a drag force. It has the expression $F_A = -\frac{1}{2} m_f \frac{dv}{dt}$
4. The drag force, F_D , caused by the fluid viscosity given by $F_D = \frac{\pi}{6} \rho_f v |v| d^2 C_d$

BEAD SETTLING

A single spherical particle is considered to fall freely along the z axis in the direction of gravitational acceleration. Wall effects and particle interactions are neglected.

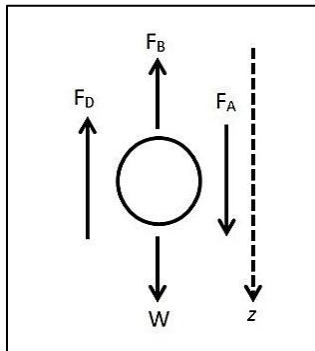


Figure 1. Forces acting on a settling spherical bead

Newton's law of motion, when applied to the spherical bead, has the form

$$ma = W - F_B - F_D - F_A$$

Substituting for the forces,

$$m \frac{dv}{dt} = mg - m_f g - \frac{\pi}{8} \rho_f v |v| d^2 C_d + \frac{1}{2} m_f \frac{dv}{dt}$$

$$\left(m + \frac{1}{2} m_f\right) \frac{dv}{dt} = (m - m_f)g - \frac{\pi}{8} \rho_f v |v| d^2 C_d$$

$$\frac{dv}{dt} = \frac{(m - m_f)g}{\left(m + \frac{1}{2} m_f\right)} - \frac{\frac{\pi}{8} \rho_f v |v| d^2 C_d}{\left(m + \frac{1}{2} m_f\right)}$$

$$\text{Substituting, } m = \frac{1}{6} \pi d^3 \rho \text{ and } m_f = \frac{1}{6} \pi d^3 \rho_f$$

$$\frac{dv}{dt} = \frac{(\rho - \rho_f)g}{\left(\rho + \frac{1}{2} \rho_f\right)} - \frac{3\rho_f v |v| C_d}{4d \left(\rho + \frac{1}{2} \rho_f\right)}$$

Rearranging,

$$\frac{dv}{dt} = \frac{1}{A} (B - C * v * |v| * C_d) \quad \text{and} \quad \frac{dz}{dt} = v$$

Where,

$$A = 1 + \frac{1}{2} \bar{\rho}, B = (1 - \bar{\rho})g, C = \frac{3\bar{\rho}}{4d}, \bar{\rho} = \frac{\rho_f}{\rho}$$

BEAD RISE

Neglecting the wall effects and particle interactions, a single spherical particle is considered to rise along the z-axis.

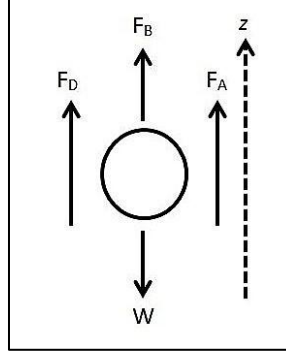


Figure 2. Forces acting on a rising spherical bead

From Newton's law of motion,

$$ma = F_B + F_D + F_A - W$$

$$m \frac{dv}{dt} = m_f g + \frac{\pi}{8} \rho_f (U - v) |U - v| d^2 C_d + \frac{1}{2} m_f \frac{dv}{dt} - mg$$

$$\left(m + \frac{1}{2} m_f\right) \frac{dv}{dt} = (m_f - m)g + \frac{\pi}{8} \rho_f (U - v) |U - v| d^2 C_d$$

$$\frac{dv}{dt} = \frac{(m_f - m)g}{\left(m + \frac{1}{2} m_f\right)} - \frac{\frac{\pi}{8} \rho_f (U - v) |U - v| d^2 C_d}{\left(m + \frac{1}{2} m_f\right)}$$

Substituting, $m = \frac{1}{6} \pi d^3 \rho$ and $m_f = \frac{1}{6} \pi d^3 \rho_f$

$$\frac{dv}{dt} = \frac{(\rho_f - \rho)g}{\left(\rho + \frac{1}{2} \rho_f\right)} - \frac{3\rho_f (U - v) |U - v| C_d}{4d \left(\rho + \frac{1}{2} \rho_f\right)}$$

Rearranging,

$$\frac{dv}{dt} = \frac{1}{A}(B + C * (U - v) * |(U - v)| * C_d) \quad \text{And} \quad \frac{dz}{dt} = v$$

Where,

$$A = 1 + \frac{1}{2}\bar{\rho}, B = (\bar{\rho} - 1)g, C = \frac{3\bar{\rho}}{4d}, \bar{\rho} = \frac{\rho_f}{\rho}$$

VITA

Bharathwaj Gopalakrishnan

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Thesis: SEPARATION OF RESIN TYPES IN MIXED BED ION EXCHANGE COLUMNS

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Date of Degree: May 2011

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Title of Study: SEPARATION OF RESIN TYPES IN MIXED BED ION EXCHANGE

Pages in Study: 83

Candidate for the Degree of Master of Science

Major Field: Chemical Engineering

Scope and Method of Study: Resin separation is a vital step prior to regeneration of Ion Exchange resins. The degree of separation determines the extent of sodium and sulfate or chloride into the system from caustic and acid regenerants, respectively. This study is aimed at determining the backwash conditions that would yield the best separation and also investigate the efficiency of interface isolation method for resin transfer.

Findings and Conclusions: Flowrate and height of bed expansion are the two important parameters controlling resin separation. Increased backwash time along with low flow leads to better separation of a mixed bed. Interface isolation method is a viable resin transfer technique. The two factors affecting transfer are pressure and operator skill

ADVISER'S APPROVAL: Dr. Gary L. Foutch
