WATER PURIFICATION USING TAM5

A Senior Thesis

By

Elizabeth Philip

1997-98 University Undergraduate Research Fellow

Texas A&M University

Group: Engineering III

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ELIZABETH PHILIP

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Abstract

Several novel crystalline silicotitantes were developed at Texas A&M University, and one of them is the 1996 R&D 100 Award winning material, TAM5. Although presently the sole use of TAM5 is to clean radioactive wastes, TAM5 has a unique structure that makes it ideal for sodium removal from water systems. The sodium levels of College Station tap water are between 300 and 450 ppm. Sodium removal was achieved by flowing tap water through a fixed bed of TAM5. Only 20 g of TAM5 are required to remove sodium from 350 ppm to less than 1 ppm for 3 L of tap water. Initial batch experiments involving sodium nitrate and lithium chloride indicate that TAM5 can separate sodium from a lithium solution. This separation is desirable for the treatment of waste from electrometal processing. TAM5 has proven effective for separating sodium under various conditions.

In comparison to literature values for a standard size Ultrapure bed, one of the most common ion exchange resins on the market, TAM5 proved superior. TAM5 can remove 11 - 20% more sodium than Ultrapure. A 20 g column of TAM5 can be regenerated using only 60 ml of 2M HCl. Thus a TAM5 column can be used in a cyclic process of sodium removal from drinking water followed by a regeneration step using an acid.

Introduction

The purification of our water systems has been a growing concern. Currently, although much research has focused on the removal of heavy metals, such as methods using live baker's yeast (1), the need for a non-heavy metal removal agent still exists. In towns (such as College Station, Texas) where the water has high sodium concentrations, current ion-exchange resins are easily exhausted and thus must be replaced frequently. Even the US Army has a need for an effective water purifier for brackish waters, of which sodium is a large component. Thus attention must be focused on an efficient and reusable sodium removal agent for drinking water.

The main objective of the experiments was to investigate the use of TAM5 as a water purification agent. TAM5, 1996 R&D 100 award-winning material, is a crystalline form of a sodiumtitanosilicate which was first synthesized at Texas A&M University in 1990(2,3). The structure of TAM5 makes it suitable for packing Cs ions, as much as 5 - 10% by weight, from radioactive wastes; the only current use for TAM5. The radioactive waste solutions contain low ppm levels of radioactive ^{137}Cs in a matrix saturated with inorganic salts of elements such as Na, K and Al. The unique features of the TAM5 structure are the existence of several layers of parallel uniform 'micro capillaries' in the crystals and its ability to sieve ions based on size. The original TAM5 has been modified (a not yet disclosed technique with a different chemical formula) to a highly crystalline material with a higher Cs ion exchange capacity in strongly basic solutions. The Department of Energy is planing to use as much as a million pounds of this material for the removal of radioactive Cs^+ from radioactive wastes that have accumulated in millions of barrels in selected locations across the country.

Currently TAM5, under the trade name IONSIV IE-911, is commercially produced and marketed by UOP (formerly United Oil Products). It is this form of TAM5 that was used in the water purification experiments. TAM5's unique structure (undisclosed) makes it ideally suited for sodium removal. Thus its use as a sodium removal agent is promising. The use of TAM5 has focused primarily on the removal of radioactive wastes; thus, currently no other research has been done in the area described above.

Procedure

Experiment 1: TAM5 column in a glass buret:

Procedure for packed fixed bed TAM5 column:

To make a TAM5 column, first glass wool (approx. 3 cm high) was placed in a glass buret (1 cm inside diameter). A glass buret is not ideal, but was the only buret available at the time, and thus used. Then utilizing slurry packing, the buret was packed with 20 g of IONSIV IE-911. Slurry packing consists of mixing deionized water with the weighed TAM5 and then pouring the heterogeneous mixture through a funnel into a buret half-filled with deionized water. Water dripped from the bottom of the buret so that the TAM5 granules would pack well without pockets of air or water. The water was then drained through the buret such that 15 ml of the water remained above the packed TAM5 bed. The column had a bed height of 23.5 cm and a column volume of 20 cubic centimeters.

The flow rate was then adjusted to 5 ml/min using a plastic stopper located at the bottom of the buret.

A. First Acid Wash for COLUMN 1:

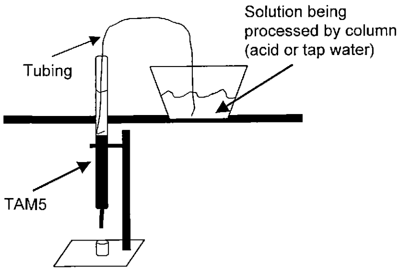

Figure 1: Siphoning setup used to provide continuous flow to the TAM5 Column

IONSIV IE-911 is the sodium-enriched form of TAM5. Therefore, since TAM5 would need to be in the hydrogen form to remove sodium from water, an acid washing procedure was necessary. The water purification column was washed with 2L of 2M HCl at 5ml/min in a setup (see figure 1) previously tested and recommended (8). The procedure used a siphoning effect to let the acid drain through the TAM5 column; a vacuum setup to speed the flow is optional. The column drained well though, so a vacuum was not necessary to speed the flow. Twenty-four samples were taken continuously during the acid wash to measure the sodium removed from the TAM5. After the acid had passed through the column, 2.5 L of deionized water was allowed to pass through the column at the same 5 ml/min flow rate. Care was taken to make sure that the column was never dry to prevent the bed from being disturbed, which would lead to a column bed with air pockets.

B. Sodium Removal from tap water:

Tap water (from the lab, Texas A&M University, College Station, TX) was processed by the column at 5mL/ min. The same type of siphoning setup (see figure 1) that was used for the acid washing procedure was used to continuously flow tap water through the TAM5 bed. Samples were taken at 250ml intervals. The samples were then diluted to within the Atomic Absorption Spectrometer's range for sodium (approx. 0 ~ 1.2 ppm). Cesium (1000 ppm in the diluted sample), in the form of Cesium nitrate, was added as an ionization suppressant, allowing a more accurate reading of sodium levels. The samples were analyzed using the Atomic Absorption Spectrometer (Varian SpectrAA30).

C. Amount of Sodium in TAM5 after tap water processing

After the water purification column processed the tap water, a 2.25 g TAM5 sample was taken from the top of the column. From this 2.25 g sample, 100 mg of TAM5 was dissolved in 1 ml HF and diluted to 10 ml with deionized water. This solution was then diluted to a final 1:1000 dilution to be analyzed by the Atomic Absorption Spectrometer. The solution also contained 1000ppm Cs as an ionization suppressant.

D. Second Acid Wash for Column 1:

The same procedure used for the first acid wash was used for the second acid wash of Column 1. Samples were taken at 100ml intervals to determine the amount of 2M HCl necessary to return the TAM5 to its hydrogen form. The samples were diluted and cesium added such that the Atomic Absorption Spectrometer could analyze them for sodium content.

E. Second Use of Column 1 to Remove Sodium from tap water:

The regenerated TAM5 column was used to process tap water from the same location as the first tap water run. Tap water was processed at a flow rate of 5 ml/min.

Samples were collected at 250 ml intervals. The samples were diluted and analyzed by the Atomic Absorption Spectrometer, as before.

Experiment 2: TAM5 column in a plastic (acrylic) buret

Two TAM5 columns were made in acrylic burets. These columns were identical in column weight, height, and volume. For the purpose of distinction one will be called COLUMN A and the other referred to as COLUMN B.

Procedure for packing TAM5 column:

To make the TAM5 column, a piece of coarse (fast flow) filter paper was cut to fit an acrylic buret (1 cm inside diameter) and placed at the bottom of the buret. Then using the slurry packing method previously described, the buret was packed with 20 g of IONSIV IE-911. The water was then drained through the buret such that 1 ml of the water remained above the packed TAM5 bed. The two columns (A & B) were prepared with this identical procedure. Column A had a bed height of 22 cm and a column volume of 20.3 ml. Column B had a bed height of 22 cm and a column volume of 20.3 ml.

A. First Acid Wash of Columns A and B

To convert TAM5 from its sodium enriched form to its hydrogen form, 500ml of 2M HCl were processed by COLUMN A and COLUMN B. The siphon apparatus previously mentioned was utilized again. The TAM5 columns processed the HCl at a flow rate of 5ml/min. Ten mililiter samples were continuously collected. The samples were diluted and then analyzed for sodium content by the Atomic Absorption Spectrometer.

B. Sodium Removal from Tap Water for Columns A and B:

College Station tap water was processed by Columns A and B. The flow rate of COLUMN A was adjusted to 5 ml/min using the plastic stopper located at the bottom of the buret. The flow rate of COLUMN B was similarly adjusted to 7.5 ml/min. Samples were taken at 250 ml intervals. The samples were then diluted and analyzed for sodium by the Atomic Absorption Spectrometer.

Experiment 3: TAM5 Column in a plastic buret with glass beads

Procedure for packing TAM5 column:

After the tap water had been processed by COLUMNS A and B, another piece of coarse (fast flow) filter paper was placed atop of the TAM5 bed. Then glass beads were washed with 2M HCl and allowed to sit in the hydrochloric acid for 20 minutes. The glass beads were then removed from the HCl solution and rinsed with deionized water. Finally, the glass beads were slurry packed to form a 2 ml volume above the TAM5 column.

A. Second Acid Wash for Columns A and B:

The columns were then washed with 70ml of 2M HCl at 5ml/min. utilizing the siphon setup, previously described. Samples were taken continuously, each measuring 2ml, during the acid wash to measure the sodium removed from the TAM5 column. After the acid had passed through the column, 0.5 L of deionized water was allowed to pass through the column at the same 5 ml/min flow rate. Care was taken to make sure that the column was never dry to prevent the bed from being disturbed.

B. Sodium Removal from Simulated Tap Water:

A simulated tap water solution was prepared to identify any differences removing sodium from a pure sodium solution would have compared to 'real' tap water. Sodium bicarbonate, 6.9153 grams, was dissolved and then diluted to 10 L with deionized water. This solution was used to simulated tap water. Then COLUMN A at 5ml/min (15 column volumes/hour) processed the simulated tap water solution. Twenty-five mililiter samples were collected continuously. Samples at 100ml intervals were diluted and then analyzed by the Atomic Absorption Spectrometer. The results were then analyzed by plotting the sodium concentration (C) divided by the initial tap water concentration (C_o = 300 ppm) versus the number of column volumes processed.

Experiment 4: Batch Experiments

For modeling purposes, batch experiments were performed for sodium and lithium solutions. To obtain the acid form TAM5 for use in the batch experiments, 20 g of IONSIV IE-911 was placed in an acrylic buret. The column at a flow rate of 5ml/min processed 120 ml of 1M HCl. Three milliliter samples were taken continuously. Analysis of these samples was performed, by Atomic Absorption, to ensure that the TAM5 was entirely in its acid form. The column then processed one liter of deionized water at 5ml/min. The TAM5 was removed from the buret and allowed to air dry completely in a weighing dish (approximately 24 hours).

A. Sodium Batch Experiments

Sodium nitrate, 9.2421 grams, was diluted to 250 ml using deionized water to form a 10,000 ppm Na solution. The 10,000 ppm Na solution was then used to make standards for Atomic Absorption Spectroscopy and solutions for use in the experiments. The 10,000 ppm solution was diluted to make 500 ml of 1000 ppm Na solution, which was used to make solutions of desired concentration for the batch experiments. Then 100 mg of TAM5 (acid form) was measured into each of forty-four 15 ml vials. Next the sodium solution and deionized water were added to make a solution of the desired concentration. A replicate for each concentration was made. The mass of the deionized water and sodium solution added was measured to correct for the deviation of sample weights from the theoretically desired weight. Solutions were made for the following concentrations (in ppm Na): 10, 50, 100, 150, 200, 250, 300, 350, 400, 450, 500, 600, 700, 800, 900, 1000. Three additional solutions were made for the 250 ppm Na and 500 ppm Na experiments. The purpose of these solutions was to ensure that equilibrium had been reach. Thus one 250 and one 500 ppm Na experiment was removed from the shaker after 24, 48, and 96 hours. The rest were removed from the shaker after 72 hours. Once removed from the shaker, the vials were allowed to rest for 4 hours. Then the supernatant liquid was decanted into a new vial and subsequently diluted for analysis using the Atomic Absorption Spectrometer. Cesium was added to the diluted samples and standards used for calibration such that the solutions contained 1000 ppm Cs. The pH of the undiluted samples were also tested using an Orion Model 730 pH meter.

B. Lithium Batch Experiments

Lithium chloride, 15.4501 grams, was diluted to 250 ml using deionized water to create a 10000 ppm Li solution. Standards for Atomic Absorption Spectroscopy were then made according to the manual. The above procedure for the sodium batch experiments was repeated for lithium. The following concentrations were tested: 10, 50, 100, 150, 200, 250, 300, 350, 400, 450, 500, 600, 650, 700, 800, 900, 1000. Diluted lithium solutions contained 1000 ppm Cs as well.

Results and Discussion

<u>Column Experiments</u>

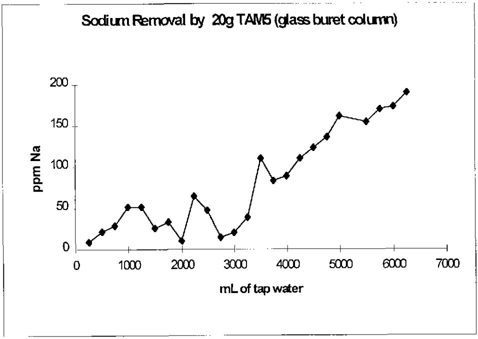

Figure 2: College Station Tap Water processed by a 20g TAM5 Column in a Glass Buret

The glass buret was not an ideal situation and thus the results were used as a general indication of the ability of TAM5 to remove sodium from drinking water. College Station tap water processed at a flow rate of 5 ml/min by a 20 g TAM5 column

(Experiment 1B) can be seen in figure 2. The initial experiment shows that TAM5 can remove sodium from real drinking water effectively. There is an obvious data scatter though. This can be accounted for by interference caused by the glass buret and glass wool.

The analysis of the TAM5 and HF solution (Experiment 1C) revealed that sodium was absorbed at approximately 4.37% by weight. Thus about 1 g of sodium can be absorbed by 20 grams of TAM5.

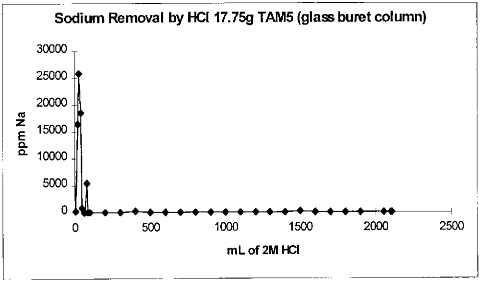

Figure 3: Regeneration of Glass Buret TAM5 Column

Figure 3 shows that the initial estimate of 2L of 2M HCl necessary to regenerate TAM5 was above the actual amount needed. Thus the amount of acid used in Experiment 2A was reduced to only 500 ml of 2M HCl.

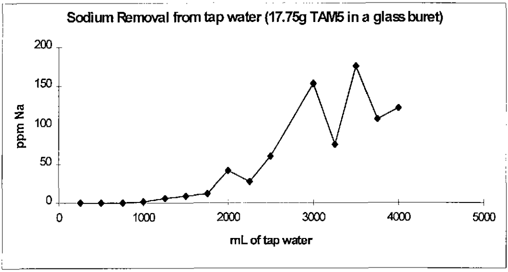

Figure 4: College Station Tap Water processed by a Regenerated TAM5 column in a Glass Buret

The ability of a column to be regenerated is the key to the success of any water purifier. Figure 4 displays the results of processing College Station tap water through a regenerated TAM5 water purification column. The column performs just as well as a newly made column. Thus a TAM5 column can be regenerated and thus reused.

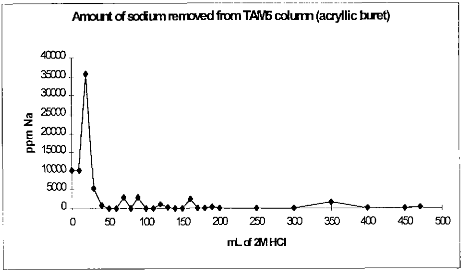

Figure 5: Converting TAM5 to hydrogen form (COLUMN A)

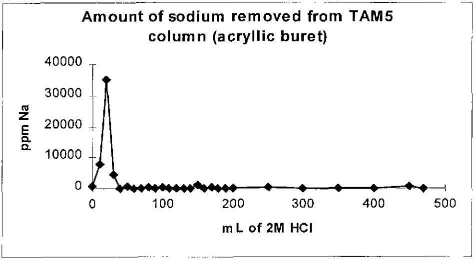

Figure 6: Converting COLUMNB to hydrogen form of TAM5

In an effort to reduce the effects of the glass buret and glass wool had on the data, the TAM5 column design was modified to bring about the design in experiment 2. In experiment 2A, we can see that the data has smoothed somewhat, but that the sodium appears to be released in plugs of high concentration (note peaks around 100 ml). This result can be accounted for as drift in the column. The theory behind the acid wash of the column is that the hydrogen in HCl and sodium in the TAM5 are exchanged. When drift occurs, as the HCl is introduced some of the sodium travels upward into the supernatant liquid while the rest travels down the column (see figure 7). Thus sodium can accumulate in the supernatant liquid and be released as high concentration plugs.

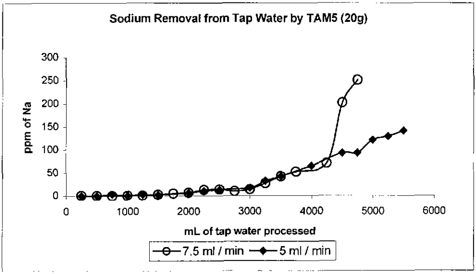

Figure 8: The Effect of Increase Flow Rate in an Acrylic TAM5 Water Purification Column

In Experiment 2B the effects of different flow rates were observed. Figure 8 shows tap water processed at 5ml/min and 7.5 ml/min by a TAM5 column. At a flow rate of 5 ml/min, it can be observed that TAM5 removed sodium to less than 1 ppm Na, for 3 L of 350 ppm Na, College Station tap water. Five milliliters per minute is a very fast flow rate for a 20 ml column. If it was desired to speed up the flow rate, at a flow rate of 7.5 ml, no difference can be seen preceding the 3 L breakthrough point. This is the only region of interest because sodium removal to a low level is desired. Both of these results are good considering that tap water is tested at levels as high as 400 ppm.

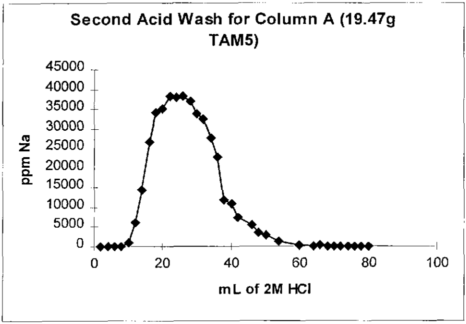

Figure 9: Regeneration of TAM5

(Acrylic Buret, COLUMN A)

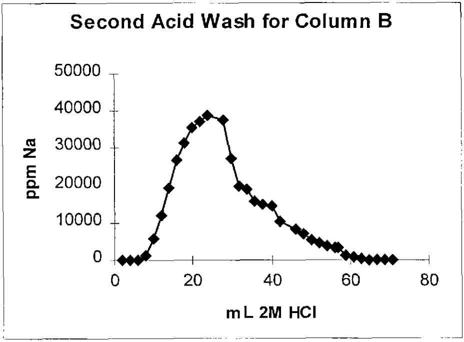

Figure 10: Regeneration of TAM5 Column

(Acrylic Buret, COLUMN B)

To reduce the drift in the column, the column design was modified. The column used a layer (~ 2 cc) of glass beads on top of the previously used TAM5 columns (A and B) from Experiment 2. The resulting regenerating acid wash curves were very smooth (see figures 10 and 11). Both figures 10 and 11 look identical, as they should for the same column material. Thus the new column design can be recommended for future experiments. Also the assumptions made concerning the glass interference and drift in the column can be validated by the results of the second acid washes of columns A and B and the simulated tap water experiment with column A.

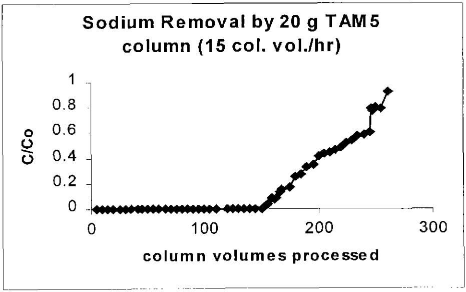

Figure 12: Simulated Tap Water (300 ppm Na)

Processed by a Regenerated TAM5 Column

Figure 12 is a plot of processed water concentration divided by the initial tap water concentration (C/Co) versus the number of column volumes processed by the TAM5 column. This plot allows a general view of TAM5 to be seen. From figure 12, it is seen that 150 column volumes can be processed to less than 1 ppm sodium in an initial tap water concentration of 300 ppm. Thus if one 2 L bottle of TAM5 was used, 300L of clean water could be obtained.

Batch Experiments

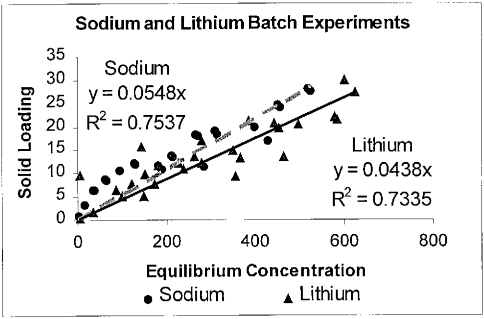

Figure 13: Kd Experiments for Sodium and Lithium

For the batch experiments plots of the solid loading versus equilibrium concentration yielded interesting results. Solid loading is defined as the difference between the initial and final concentration times the liters of solution in the vial divide by the grams of TAM5 used. Figure 13 shows the results for the sodium batch experiments. The data points resemble a two step function. The results of the lithium experiment show a more linear data set. The scatter is higher for the lithium compared to the sodium, though. Since modeling a two step function is difficult, linear approximations were used. The linear approximation yielded exciting results. The ratio of the slope of the two batch experiment lines yields the selectivity. The ratio of the slopes of the lithium to sodium least squares lines results in a value of 1.251. This indicates that in a solution containing sodium and lithium, sodium would be 'preferred' by TAM5. The solutions used in lithium manufacturers and electrometal processing industries have high concentrations of lithium (~70% Li and 30% Na solution), in instances such as these the ratio would be approximately 2.25. Thus a separation could be achieved.

Data Comparison to the Theory

The theory behind flow through a fixed bed column predicts a Gaussian function for plot of the concentration of an ion in the final solution versus the amount of water processed by the column. When plotted on a log probability scale (using Origin software) a Gaussian distribution becomes linear. Thus when data are plotted on a log probability plot, its deviation from the theory can be seen clearly. In reality we expect deviations from the theory. One such expected deviation is the effect of mass transfer within the TAM5 granule. Mass transfer within the granule only becomes significant when the outer 'sodium sites', on the TAM5 granule, get filled with sodium. Figures 14 through 18 (included in the Appendices) show the results of log probability plots for the column experiments. As shown in figure 14, though linearity can be seen on its entirety, a large data scatter exists. The data from the second tap water purification for the glass buret column is shown in

figure 15. The scatter in the data decreases dramatically and linearity can be seen from the fourth to the eightieth percentile. The results of the column experiments performed in acrylic burets can be seen in figures 16 and 17. Figure 16 shows the standard 5ml/min flow rate while figure 17 shows a column with a flow rate of 7.5 ml/min. Linearity for the 5ml/min flow rate column can be seen between the 0.02 to the eightieth percentiles. Thus it approximates the ideal condition very closely. At the faster flow rate, though, non-linearity can be observed; the deviation around 100 – 150 can be seen distinctly. The faster flow rate magnifies the non-ideality of this region previously mentioned. Comparison between the log probability plots for the glass and acrylic buret show that the glass buret did have an effect on the data, and that an acrylic buret should be used to house the TAM5 column. Finally, the results from the purification of the synthetic tap water containing 300 ppm sodium carbonate can be seen in figure 18. From the plot, linearity can be seen between the twentieth and seventieth percentiles. The tails of the data deviate from the expected linear curve, possibly due to intre-granular transport.

Conclusions

As the results indicate, the use of TAM5 as a water purifier/sodium remover is feasible and desirable. TAM5 water purification columns can clean 150 column volumes of tap water to less than 1 ppm Na, while a 20 g column can be regenerated with only 60 ml of 2M HCl. The US Army could thus use TAM5 columns for mobile units as a portable brackish water purifier, due to the ability of TAM5 to remove sodium efficiently and reusability. From the above results it can be calculated that one gallon of TAM5 can clean enough water for one person for 259 days. Alternatively, a hundred person regiment could be supplied with 1.5 days of clean water with only a one gallon column of TAM5. Homes and laboratories would also benefit from using TAM5 columns to remove sodium from their water. According to the Thomas Scientific Catalog (96/97), a single Ultrapure bed can absorb 44.89 grams of NaCl. TAM5 is 11% more effective as Ultrapure, and thus more desirable. TAM5 in reality, though, is more than just 11% as effective as Ultrapure. Ion exchange efficiency increases as the column bed increases in size. Since the TAM5 columns were only 20 cubic centimeters, as the TAM5 bed approaches the size of standard Ultrapure beds TAM5 will be 20% better than Ultrapure.

Additionally, batch experiments indicate that sodium can be separated from lithium in solution using TAM5 columns. The ability of TAM5 to separate sodium from lithium solutions has the potential for a broad range of applications. The electroplating industries as well as lithium manufacturers are in search of an agent to accomplish this separation. TAM5 has proven effective for several separations. TAM5 water purification columns can be used in a cyclic process. After cleaning water, TAM5 can be regenerated, and thus reused to purify water once again. Therefore, the use of TAM5 as a sodium removal and water purification agent is recommended.

Future Research

One of the main objectives of further research will be to discover how long a column can be reused without damaging the TAM5 material. Although to date the column has not been known to deteriorate during column studies, more work is necessary. Also under investigation will be TAM5's ability to remove other metals (heavy and non-heavy) while in the presence of sodium. Further research is still needed to determine the efficiency of sodium removal in lithium solutions. Column modeling of sodium removal from tap water as well as the modeling of the acid wash data are areas in which investigation would be beneficial.

Acknowledgements

The assistance of Dr. R. G. Anthony is greatly appreciated. In addition to laboratory and equipment access, he provided numerous suggestions, which are greatly appreciated. A thank you also to Ph.D. graduate students Hamad Al-Adwani, Iqbal Latheef, and Michael Huckman who shared equipment and experience with me. Finally, the efforts of Dr. L. S. Fletcher with the Offshore Technology Research Center in conjunction with the National Science Foundation as well as the Texas A&M University Undergraduate Fellows are acknowledged for their support of this research.

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Appendix: Log Probability Plots

Figure 14: College Station Tap Water processed by a 20 g TAM5 Column in a Glass Buret (15 column volumes/hour)

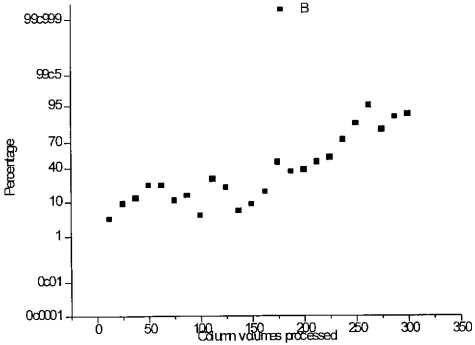

Figure 15: College Station Tap Water processed by a Regenerated TAM5 Column in a Glass Buret (15 column volumes/hour)

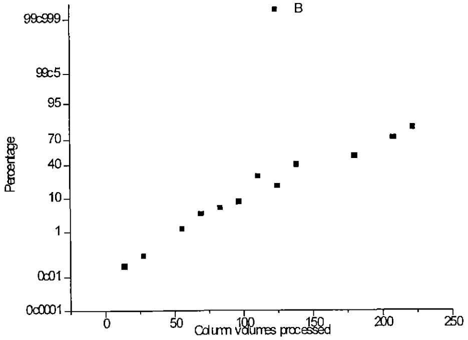

Figure 16: College Station Tap Water processed by 20 g TAM5 in an Acrylic Buret (COLUMN A, 15 column volumes/hour)

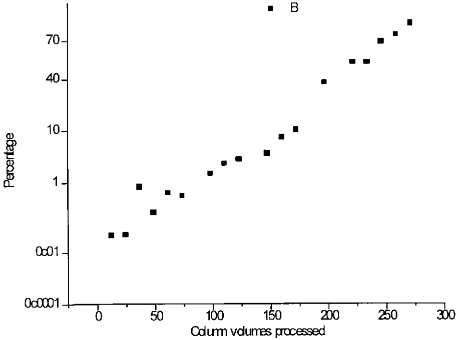

Figure 17: College Station Tap Water processed by 20 g TAM5 in an Acrylic Buret (COLUMNB, 22.5 column volumes/hour)

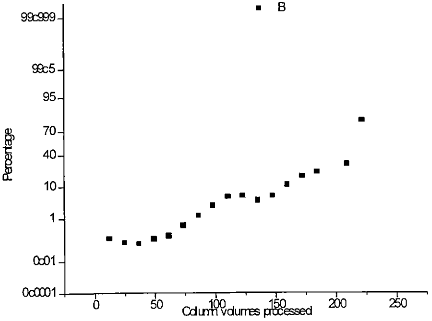

Figure 18: Synthetic Tap Water (300 ppm Na) processed by Regenerated TAM5 at 15 column volumes/hour