

Novel Methods of Brine Concentration

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ABSTRACT

A study was made of unconventional methods for concentrating the waste effluents from desalination plants to facilitate their disposal. Several of these could find application as primary desalting processes, with the unique feature that only very concentrated waste effluents are produced.

The methods reviewed include solvent extraction by paraffinic hydrocarbons at high temperatures, solvent extraction by tertiary amines at ambient temperatures, various ion exchange schemes, foam concentration, and other miscellaneous processes. Preliminary flowsheets, material balances, and cost estimates were prepared for these processes, assuming various saline brines.

It was concluded that ion exchange processes which include production or regenerants from the dissolved solids offer the lowest costs of the methods investigated for concentrating waste brines which contain less than about 7000 ppm total dissolved solids. (TDS). Above that level solvent extraction schemes are more economical.

INTRODUCTION

The U.S. Department of the Interior, Office of Saline Water, has sponsored work in membrane desalination for conversion of inland brackish waters to potable water. This work has progressed to the point where commercial plants, are providing municipal and industrial water supplies from brackish water sources. The problem of the economic disposal of concentrated effluents at these inland locations is critical.

This report concludes a study by Garrett Research and Development Company into methods for concentration of waste effluents from membrane desalting plants to brines containing 20% dissolved solids (Battey et al., 1971). The specific processes to be investigated were two-component solvent extraction of salts, high temperature solvent ex-

traction of water, ion exchange and electrolysis, solvent precipitation and foam concentration. Typical parameters considered were processing characteristics, pre-treatment and energy requirements, chemical consumption, materials of construction, and capital and operating costs of the system. The systems proposed are independent of the desalting plant; that is, the composition of the intermediate effluent brine was not a variable under study.

The above methods are not commonly used for concentrating saline brines. There are, however, several additional methods of brine concentration which are more or less common practice. Among these are electrodialysis, reverse osmosis, freezing, distillation, and solar evaporation. The brine concentration methods which were studied can, in some circumstances, be used for concentrating brines for production of fresh water from saline brines and for the production of chemicals as well as for the disposal of waste saline brines.

The various processes considered were evaluated for each of four saline brines. These brine compositions are typical of inland saline brines and they are listed in Table I. The processes were developed by first developing a process flowsheet and material balances for each brine. Capi-

TABLE I
Composition of Four Brines

Chemical Constituent (p.p.m.)	No. 1	No. 2	No. 3	No. 4
Calcium	395	1,400	785	492
Magnesium	175	940	343	315
Sodium	265	3,400	1,125	3,404
Potassium	34	91	—	39
Sulfate	2,329	2,580	3,030	8,680
Chloride	26	7,850	1,870	185
Bicarbonate	21	1,150	51	1,153
T.D.S.	3,245	17,411	7,204	14,468

tal and operating costs were estimated for government financed desalination projects (30 year amortization and 4-5/8% interest rates). Each material balance was based on concentrating the brine to 20% total dissolved solids.

HIGH TEMPERATURE SOLVENT EXTRACTION

This process uses C-11 and C-12 paraffinic hydrocarbons to extract water from a saline brine. At a temperature of 650°F and 2,600 psig, the solubility of water in the hydrocarbon is approximately 80 mole percent, while the dissolved solids are practically insoluble in the hydrocarbon.

P. Barton and M. R. Fenske (1970) have conducted an extensive investigation of the feasibility of solvent extraction of saline brines for desalination. They have concluded that the process described above, using C-11 and C-12 hydrocarbons, appeared the most feasible and presented a design for a 10 mgd desalination plant (Barton and Fenske, 1970). This design is based on solubility data (Guerrant, 1964) and liquid-solids heat exchanger data (Kopko, 1969). They concluded that sea water could be converted to fresh water at a cost of about \$1.00/1,000 gal. of fresh water in their 10 mgd plant.

A one mgd waste brine disposal plant was designed which is similar to the 10 mgd plant design (Barton and Fenske, 1970). The output of the waste brine disposal plant consists of brine (20% wt. dissolved solids) and fresh water (120 ppm dissolved solids, 12 ppm dissolved hydrocarbons).

The plant utilizes raining solids heat exchangers of the type designed by Barton and Fenske. The spherical alumina solids (3/8 in. dia.) are fed through the exchangers by gravity and elevated by entrainment with a liquid. The plant being considered uses four of these exchanger pairs. Figure 1 is a diagram of the plant considered.

The total installed cost of all equipment is given in Table II. Equipment costs are presented for units to process brines 1 and 2. Table II also includes an itemization of the cost of the brine disposal. Operating costs, labor, and amortization are included. The product fresh water may be sold for 30¢/1,000 gal. The resulting cost of the brine disposal will then be \$1.15-\$1.19/1,000 gal. If this is to be considered as a process for producing fresh water from a brine, the cost of the fresh water would be \$1.48/1,000 gal. if produced from brine 1 and \$1.60 if produced from brine 2.

The cost of disposing of brine 1 is nearly equal to the cost of disposing of brine 2. Since brines 3 and 4 have dissolved solids concentrations which lie between those of 1 and 2, it may be assumed that the cost of disposing of brines 3 and 4 will lie between \$1.15 and \$1.19/1,000 gal. of brine.

The cost of concentrating an effluent brine by high

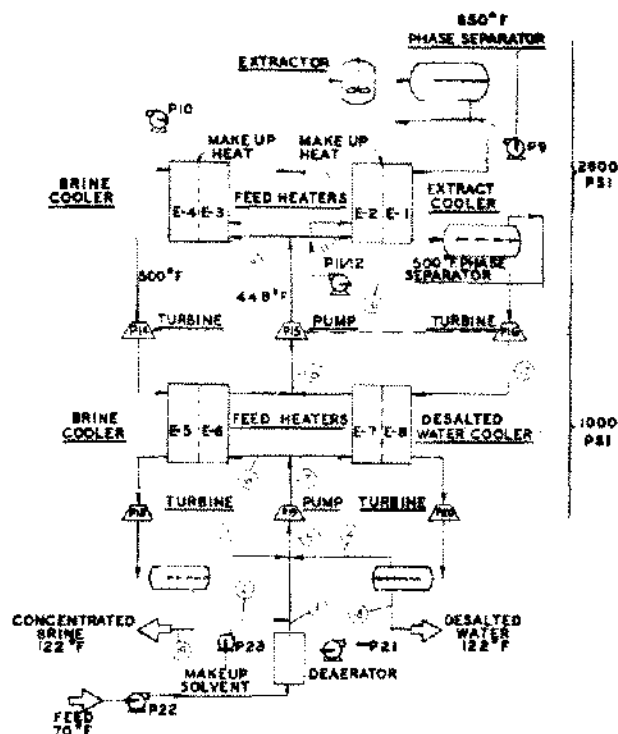


Figure 1. Schematic flow diagram of the high temperature solvent extraction process.

TABLE II

High temperature solvent extraction equipment and operating costs

Equipment Costs	Brine #1	Brine #2
Principle Items of Equipment (installed)	\$1,343,000	\$1,362,000
Total Plant Investment	\$3,296,000	\$3,342,000
Working Capital (60 days of operation)	\$ 95,000	\$ 96,000
Startup Capital (30 days of operation)	\$ 48,000	\$ 49,000
Investment Cost/gpd of Disposal Components	\$ 3.10	\$ 3.14
Operating Costs Per Stream Day		
Fuel and Power	\$ 417.00	\$ 414.00
Amortization	\$ 622.00	\$ 631.00
Other Operating Costs	\$ 570.00	\$ 577.00
Total Costs	\$ 1,609.00	\$ 1,622.00
Credit for Fresh Water (30¢/1000 gal.)	(\$ 327.00)	(\$ 304.00)
Cost Per 1000 gal. of Waste Brine	\$ 1.15	\$ 1.19

Basis: 1,000,000 gpd, 0.9 Stream Factor Costs as of September 1970

temperature solvent extraction is relatively independent of the composition of the brine. Since this process results in a potable water product, it may be used directly for desali-

nation of saline water as well as for concentrating the effluent of other desalting plants.

LOW TEMPERATURE SOLVENT EXTRACTION

The solvent extraction of water from a saline brine at ambient temperature has been investigated for possible use in desalination by Hood, Davidson, and others at Texas A&M College (Giona, 1964; Hood and Davison, 1960). These investigators concluded that tertiary amines had a potential for use in desalination by solvent extraction (Davison et al., 1960, 1966; Texas A&M, 1961). Initial economic evaluations indicated that solvent extraction at or near ambient temperature was competitive with other types of desalination.

A pilot plant investigation was completed in 1967 with the conclusion that the process operated satisfactorily although solvent losses were high. A subsequent laboratory investigation of solvent losses indicated that these could be kept below 11%/yr. (Davison et al., 1968). Typical conditions investigated in the pilot plant study were: Feed water containing 4,000 to 9,000 ppm salt, concentrated brines of 1.5 to 8% salt, product water of 10 to 600 ppm salt, extract water content of 30%, and recycled solvent of about 6% water.

The Texas A&M low temperature solvent extraction process possibly may be used to concentrate effluent brines to 20% by weight total dissolved solids (TDS). Their pilot plant operations resulted in a maximum raffinate concentration of 8% TDS; however, higher concentrations may be possible. Figure 2 is a diagram of the low temperature solvent extractions process. Water is relatively soluble in the amine at 91°F and relatively insoluble at 140°F.

The extractant containing a considerable amount of water leaves the extraction column and flows through the heat exchange system in two streams. One exchanges heat with stripped solvent, and the other with separated water. The extractant is further heated to the separation temperature of 140°F. Stripped solvent is cooled by extract and then by cooling water back to 91.4°F and fed to the extraction column. Water from the high temperature phase separator flows through the extraction reflux.

The equilibrium data obtained by the Texas A&M investigators indicate that for brine 1, seven theoretical stages are necessary for the countercurrent extraction. It is not known how accurate the equilibrium data are at high salt concentrations.

The process in Figure 2 includes a furnace to heat water. In an actual operation, a more inexpensive heat source may be found since heat is required at a low temperature (140°F). In addition, cooling water is used to remove heat from the system. The entire process may be made to operate at a higher or lower temperature by varying the composition of the amine solvent. The process

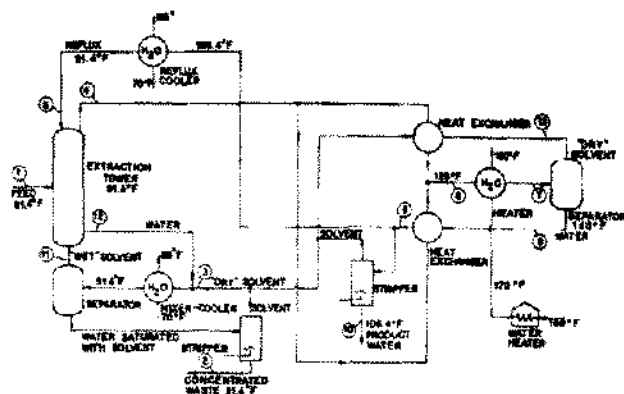


Figure 2. Schematic flow diagram of the low temperature solvent extraction process.

illustrated in Figure 2 contains a solvent which is half methyldiethylamine and half triethylamine.

A material balance and cost estimate was made for brine 1 only. The feed contains 3,245 ppm TDS, the product water 500 ppm TDS, and the concentrated waste 200,000 ppm TDS. The outlet stream contains less than 1.0 ppm dissolved amines.

The Principle Item of Equipment costs listed in Table III are based on published cost data for heat exchangers, extraction columns, etc. These costs have been updated to September, 1970.

The operating costs are also listed in Table III. Because heat is required at the low temperature of 140°F, a furnace efficiency of 100% is assumed. Solvent losses are assumed to be 11%/yr. of the 273,000 lb. inventory (50¢/lb.) and cooling water was charged at a rate of 1.5¢/1,000 gal. Approximately 15% of the product water is used as makeup for the cooling system.

The cost of disposing of 1,000 gal. of brine 1 is \$1.16.

TABLE III

Low temperature solvent extraction equipment and operating costs

Equipment Costs

Principle Items of Equipment (installed)	\$1,049,000
Total Plant Investment	\$2,764,000
Startup and Working Capital	\$ 140,000
Investment Cost/gpd of Disposal Capacity	\$ 2.61

Operating Costs Per Stream Day

Fuel and Power	\$ 424.00
Amortization	\$ 496.00
Other Operating Costs	\$ 652.00
Total Operating Costs	\$ 1,572.00

Credit for Fresh Water	(\$ 284.00)
Disposal Cost Per 1000 gal. of Waste Brine	\$ 1.16

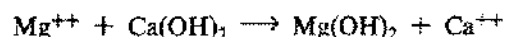
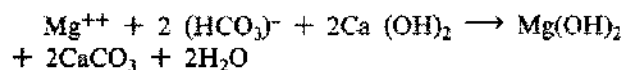
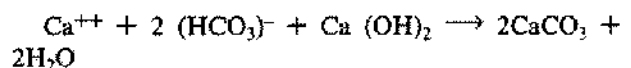
Basis: 1,000,000 gpd, 0.9 Stream Factor, Brine No. 1.

The cost of disposing of brines 2, 3, and 4 could be expected to be slightly higher as was the case with high temperature solvent extraction. The higher cost for these brines would result largely from a smaller credit for potable water.

ION EXCHANGE PROCESSES

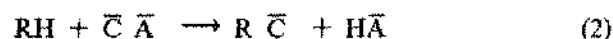
Preliminary cost estimates for three methods of concentrating saline brines using ion exchange were completed. Since high chemical costs usually prohibit the use of ion exchange on water with more than 1,000 ppm TDS, it was decided to incorporate into the designs methods for producing several of the required chemicals. The raw materials used for the production of these required chemicals in certain cases may be removed from the saline brine being concentrated. This concept utilizes the value of the dissolved solids present in the saline brine. The value of waste brines has been discussed by various authors (Leiserson and Scott, 1969; Neuman, 1967; Salutsky, 1965, 1966; Christensen et al., 1967; Tallmadge, 1964). They concluded that sea water and other brines possessed dissolved solids of sufficient value to warrant their recovery.

Dissolved calcium and magnesium are commonly removed from solution by the addition of lime and soda ash which results in the precipitation of calcium and magnesium compounds. This may be done under hot or cold conditions and is represented by the following series of reactions:



It is evident that this procedure (lime soda softening) does not reduce the equivalent quantity of dissolved solids; however, it is commonly done to remove hardness (i.e., multivalent cations). It also is evident that this is an expensive process since at least stoichiometric quantities of lime and soda ash must be added.

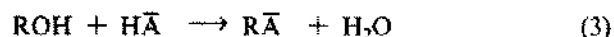
Ion exchange is a process which uses an insoluble resin which bonds chemically with cations in the case of cation exchange resins, or with anions in the case of base exchange resins. A typical reaction for cation exchange resin would be:



where C represents a cation, A represents an anion, and R the resin. In the above case, the acidic resin is introduced into a solution of dissolved salts; the solution becomes acidic and the resin bonds to the cations. The

above reaction will occur at a certain kinetic rate and reach an equilibrium. By using countercurrent flow of the solution and the resin, the saturation limits may be closely approached.

A combination of cation and basic resins may be used to completely deionize water. This is normally done by first subjecting the solution to the above acidifying exchange. This is followed by a basic exchange:



Thus, water may be exchanged for a dissolved salt (Bregman and Shackelford, 1969). In the above cases, the resins are subjected to the reverse reactions for regeneration.

The hydrochloric acid and caustic soda required for regeneration of deionization resins may be produced electrolytically from sodium chloride by the following reaction:

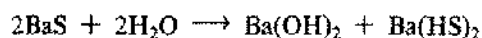
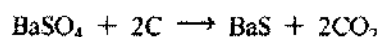
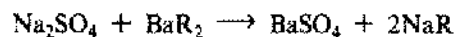


This reaction is usually accomplished with concentrated brines in either a diaphragm cell or a mercury cell.

The hydrogen and chlorine gases may be burned and dissolved in water to produce a hydrochloric acid solution for ion exchange regeneration.

This method of producing caustic soda and hydrochloric acid has been suggested (Blanco et al., 1967) for removing calcium from the feed of a multistage flash distillation plant. Carbonated caustic soda is added to the feed to precipitate calcium carbonate. The hydrochloric acid is added to return the pH to 7.0; excess hydrogen and chlorine are to be sold. The sodium chloride is prepared from the effluent of the desalting plant.

The Bureau of Mines has developed a process for removing sulfate salts from saline brines (George et al., 1967). This process was suggested as a means of producing sulfur and soda ash from Great Salt Lake brines. A barium-sodium ion exchange is used to replace cations in the solution with barium. The barium then precipitates as barium sulfate. The sulfate is mixed with coke (low cost coke with a high sulfur content possibly may be used), dried, pelletized, and roasted. The roasting produces barium sulfide which is leached to form a solution of barium hydroxide and barium hydrosulfide. This solution then is used to regenerate the ion exchange resin. The effluent sodium hydroxide and sodium hydrosulfide are carbonated to yield hydrogen sulfide and sodium bicarbonate. The hydrogen sulfide is burned to sulfur or sulfuric acid and the bicarbonate is calcined to yield soda ash. The above process may be represented by the following:



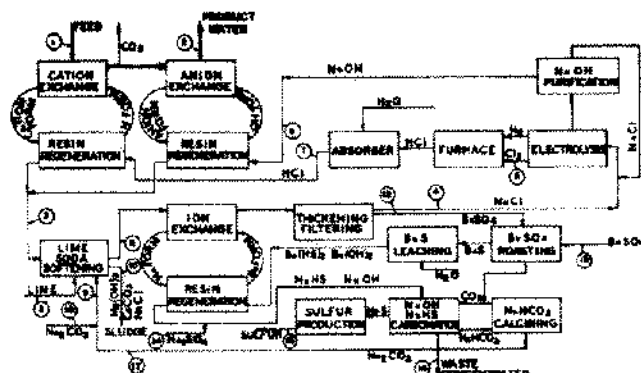


Figure 4. Diagram of process for brine concentration using dionization with HCl and NaOH regeneration.

since they may be economically concentrated by the lime-soda water softening-desulfation method. However, material balances and cost estimates were prepared for brines 2 and 3. Again, credit is given for the sale of product water and sulfur, with no charge for disposal of concentrated waste streams. The capital and operating cost estimates presented in Table V indicate that the disposal costs will be \$5.06 and \$2.31 per 1,000 gal. of feed, for brines 2 and 3.

In general, hydrochloric acid is not used for ion exchange regeneration since it is much more expensive than the more commonly used sulfuric acid. For this reason, the above method was modified to include the production of sulfuric acid from sulfur.

TABLE V

Deionization with HCl and NaOH regeneration equipment and operating costs

Equipment Costs	Brine #2	Brine #3
Total Plant Investment	\$10,296,000	\$5,477,000
Working Capital and Startup Costs	\$ 532,000	\$ 261,000
Investment Cost/gpd of Disposal Capacity	\$ 9.74	\$ 5.16
Operating Costs Per Stream Day		
Fuel and Power	\$ 984.00	\$ 421.00
Chemicals	\$ 1,114.00	\$ 506.00
Amortization	\$ 1,944.00	\$ 1,034.00
Other Costs	\$ 1,933.00	\$ 970.00
Total Costs	\$ 5,975.00	\$ 2,931.00
Credit for Fresh Water	(\$ 333.00)	(\$ 333.00)
Credit for Sale of Sulfur	(\$ 19.00)	(\$ 28.00)
Cost of Disposal Per 1,000 gallons	\$ 5.06	\$ 2.31

Basis: 1,000,000 gpd, 0.9 Stream Factor

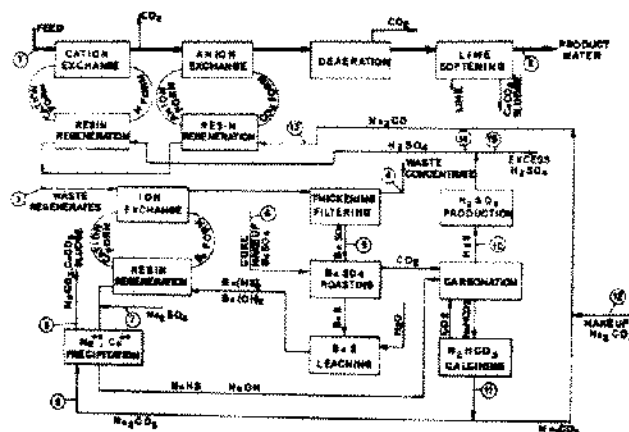


Figure 5. Diagram of process for brine concentration using dionization with H_2SO_4 and Na_2CO_3 regeneration.

Conventional deionization with sulfuric acid and soda ash regeneration

The process illustrated in Figure 5 is similar to the previous process. However, in this case, the ion exchange resins are regenerated with soda ash and sulfuric acid. The sulfate ions are removed from the waste regenerant stream by the desulfation process. The sulfur product is converted to sulfuric acid which is used for the regeneration of the cation exchange resin (excess sulfuric acid is sold). The soda ash produced is used to precipitate calcium and magnesium and to regenerate the weak base ion exchange resin. Additional soda ash is required. Aeration of the product water is required to remove dissolved carbon dioxide. Additional lime neutralization may be required to remove the last traces of this dissolved gas.

For convenience, the sulfuric acid production and capital costs were itemized separately in the operating and capital cost estimates. Credit was allowed for the sale of product water at 30¢ per 1,000 gal. and for the sale of sulfuric acid. Table VI contains the resulting capital and operating cost estimates. The disposal cost of brines 2 and 3 will be \$3.16 and \$1.38 per 1,000 gal. of feed.

MISCELLANEOUS PROCESSES

Foam concentration

Ions can be removed from a solution by use of ion adsorption on the surface of bubbles. Gas is bubbled up through a vertical column containing a surface-active liquid solution and relatively stable bubbles are formed. The solute is adsorbed on the surfaces of the rising bubbles and carried upwards to the top of the column where the bubbles break, releasing the solute and surfactant.

A foam fractionation column can be operated in several modes (Lemlich, 1968). For the large changes in concentration required in a brine disposal process (greater than

TABLE VI

Deionization with H_2SO_4 and Na_2CO_3 regeneration

Equipment Costs	Brine #2	Brine #3
Total Plant Investment	\$3,013,000	\$2,127,000
Working Capital and Startup Costs	\$ 303,000	\$ 157,000
Investment Cost/gpd of Disposal Capacity	\$ 2.98	\$ 2.06
Operating Costs Per Stream Day		
Fuel and Power	\$ 208.00	\$ 112.00
Chemicals	\$ 2,067.00	\$ 938.00
Amortization	\$ 569.00	\$ 402.00
Other Costs	\$ 1,422.00	\$ 934.00
Total Costs	\$ 4,266.00	\$ 2,386.00
Credit for Sale of Water	(\$ 333.00)	(\$ 333.00)
Sale of Sulfuric Acid (\$35/ton)	(\$ 424.00)	(\$ 525.00)
Cost Per 1,000 gal. of Waste Brine	\$ 3.16	\$ 1.38

Basis: 1,000,000 gpd Average Feed Rate

20% TDS in the concentrated brine and less than 500 ppm TDS in the potable water) a column combining enriching and stripping sections would be used. Figure 6 shows such a fractionation column where the feed enters midway in the column and reflux, concentrated in solute, is returned to the top. Stripped water is withdrawn from the bottom pool. Air is introduced into the pool to generate the foam. Sufficient surfactant is added to the pool to maintain the foam throughout the column.

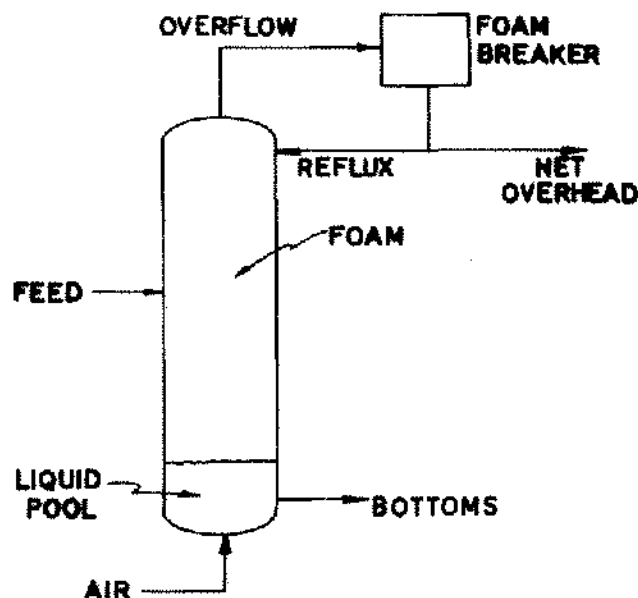


Figure 6. Diagram of column for brine concentration using foam fractionation.

Investigators of foam-ion removal have, to date, been interested only in the removal of a particular ion from a solution and have not been concerned about the ion displaced from the surfactant molecule that remains in the solution after the exchange occurs. If the removal of all ions from solution were the aim, then only hydrogen or hydroxyl ions may be exchanged for the unwanted ions. Most investigators (Bikerman, 1970) use the salts of surfactants since they are more soluble, more available and expensive. This means that there is little or no data available for process design of a possible brine concentration method.

The procedure described by Lemlich (1968) was used to estimate the required air rates. For the most dilute brine approximately \$28/1000 gal. would be required for compressor power costs alone. Air rates and power costs such as these are completely unreasonable for further consideration. Foam concentration is suited only for removing relatively small amounts of inorganic contaminants, and seems to be particularly suited to the selective concentration of specific ionic species.

Solvent precipitation and extraction of salts

One interpretation of the term "solvent precipitation" is the addition of another component (a solvent) to a brine system to result in the precipitation of some of the dissolved solids. This has been applied to nearly saturated brines for selective removal of Na_2SO_4 (Gaska and Goode-nough, 1966), but it is not suitable for concentration of brines in the 3,000 to 17,000 ppm TDS range.

Work (Davis and Grinstead, 1970) on mixed ionic (or two-component) extraction systems for by-product recovery from brines has led to the conclusion that these systems primarily separate rather than concentrate ionic species. Additionally, moderate amounts of fresh water are required for stripping, and this decreases the attractiveness for areas where desalination is employed. Chemical stripping, or regeneration, offers the promise of better process economics and is currently being investigated by others.

The use of chelation as a means of removing ions from sea water was reviewed (Lacy, 1961). Methods considered included using a water soluble chelating agent to form solvent-extractable chelates and using a water-immiscible liquid chelating agent. Because of the very high price of chelating agents and reasonable estimates of losses, these methods were judged uneconomical.

Other processes

A considerable amount of work has been reported on electrically induced adsorption on carbon electrodes (Evans and Hamilton, 1966; Murphy and Tucker, 1966). Other investigators have classified this process as a type of ion exchange (Accomazzo, 1969; Evans et al., 1969). This involves the migration of ions to usually porous carbon

electrodes under the influence of a direct current electric field. Periodically the polarity is reversed and NaCl is desorbed into the water flowing through the cell. Difficulties have been encountered particularly with electrode materials having the proper degree of porosity and electrical conductance. In addition, the process appears capable only of small concentration changes in quite dilute brines. Methods such as magnetic field separation (Gilliland, 1967), differential redox desalination (Onstott, 1964), and thermal diffusion (Shinnar and Passy, 1963) are at a very early stage of development. That is, little more than a possible phenomenon has been demonstrated. More experimental work is necessary to demonstrate any applicability to brine concentration, and these could not be considered even for preliminary economic analysis.

CONCLUSIONS

1. The ion exchange processes, which include production of regenerants from the dissolved solids, offer the lowest costs of the methods investigated for concentrating desalting plant effluents below about 7,000 ppm TDS. Above that level solvent extraction is more economical. See Figure 7.
2. The cost of concentrating a brine by ion exchange depends on the composition of the brine with respect to both total dissolved solids and chemical makeup. In general, a brine with a higher dissolved solids content will require a higher disposal cost.
3. The cost of brine concentration by both solvent extraction methods is relatively independent of the composi-

tion of the brine. Therefore, these methods are more advantageous for processing more concentrated brines.

4. The ion exchange and solvent extraction processes considered all produce potable water and thus may be considered as direct desalination processes.

5. The softening-desulfating ion exchange process is capable of producing potable water from a low chloride brine on a mgd scale at a cost of 50–65¢/1,000 gal.

Conversion Factors

English	Metric
Psi	0.0703 Kg/cm ²
\$1.00/1000 gal.	\$0.264 2/m ³
1,000,000 gal/day	3785 m ³ /day
Ton	0.9072 tons (metric)
Inch	2.54 cm.
°F	°C = $\frac{°F - 32}{1.8}$

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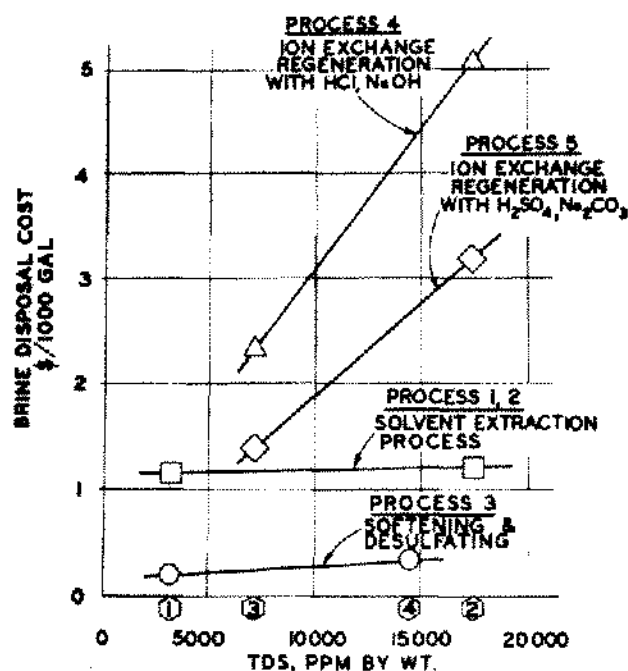


Figure 7. Comparative brine disposal cost.

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