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ENERGY CONSERVATION BY HYPERFILTRATION

Food Industry Background Literature Survey

April 15, 1980

Work Performed Under Contract No. AC03-79CS40264

CARRE, Inc.
Conservation and Resource Recovery Engineering
Seneca, South Carolina

U. S. DEPARTMENT OF ENERGY

Division of Industrial Energy Conservation

NOZUJIKUHI

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ENERGY CONSERVATION
BY
HYPERFILTRATION

FOOD INDUSTRY
BACKGROUND LITERATURE SURVEY

Prepared for
National Food Processors Association
Department of Energy Contract
No. DE-AC-03-79CS40264

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April 15, 1980

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CHAPTER 1

CONCLUSIONS AND RECOMMENDATIONS

The application of hyperfiltration to selected food product streams and food processing wastewaters for energy conservation are of concern in this project. This literature survey had led to the following conclusions and recommendations.

CONCLUSIONS

- 1) No research has been conducted in the food industry using membranes with hot process streams due to the temperature limitation ($<40^{\circ}\text{C}$) of the typically studied cellulose acetate membranes.
- 2) Based on the bench-scale research reviewed, concentration of fruit and vegetable juices with membranes appears to be technically feasible.
- 3) Pretreatment and product recovery research was conducted with membranes on citrus peel oil, potato processing and brine wastewaters and wheys. The experiments demonstrated that these applications are feasible.
- 4) Many of the problems that have been identified with membranes are associated with either the suspended solids or the high osmotic pressure and viscosity of many foods.
- 5) Research using dynamic membranes has been conducted with various effluents, at temperatures to $\sim 100^{\circ}\text{C}$, at pressures to 1200 psi and with suspended solids to $\sim 2\%$.
- 6) The dynamic membrane is being prototype tested by NASA for high temperature processing of shower water.

RECOMMENDATIONS

The literature review substantiates potential for dynamic membrane on porous stainless tubes to process a number of hot process and effluent streams in the food processing industry. Hot water for recycle and product concentrations are major areas with potential for economic

application. The two plants involved in the first phase of the project should be reviewed to identify potential energy conservation applications. Other conservation applications, not represented at these two plants, should then be listed. In particular seafood processing is not represented at these sites. As many as possible of the conservation applications should be tested during the screening phase at each site, within time limitations of Phase I. The most promising applications at each site should be evaluated more intensively to establish engineering estimates of the economics of this technology for canned fruit and vegetable segment of the food industry.

CHAPTER II

INTRODUCTION

BACKGROUND

Much energy is used to process food. The quantity of energy discharged as hot industrial process water today by the food processing industry can be considered a valuable economic resource. Much of the water used in the processing of food is hot, and if recycled back to processing would provide both energy and water conservation. If contaminants were concentrated by membranes the efficiency of conventional treatment of the pollutants may also be increased. Therefore, the benefits of recovering and reusing industrial process water are more than just the value of the water and energy alone.

This project was developed to evaluate the potential application of high temperature hyperfiltration (HF) in the food industry. Specifically, two industry groups will be considered: 1) the canned fruits and vegetables industry, and 2) the specialties food processing industry. The principal membrane application areas that are being considered are: 1) hot process recycle water, e.g., purification for direct water recycle, 2) preconcentration, e.g., initial concentration of tomato juice prior to additional concentration by phase change, 3) pretreatment of waste water, e.g., removal of pollutants to reduce treatment costs and 4) recovery of process materials, e.g., lye peeling chemicals.

The membranes that will be used are dynamic hyperfiltration membranes capable of withstanding high temperature ($>90^{\circ}\text{C}$) liquids. This hyperfiltration membrane (Zr(IV) oxide polyacrylate) differs from other hyperfiltration (reverse osmosis) membranes in that it is capable of high temperature operation.

The project is divided into four phases:

- Phase I - Evaluation of the literature and related R&D projects and preliminary visits to candidate plants
- Phase II - On site pilot-scale testing at Site 1

Phase III - On site pilot-scale testing at Site 2

Phase IV - Data evaluation and reporting

This report is the evaluation of related literature and R&D projects on membrane application in the food industry.

OBJECTIVE

The project objective is to determine the technical efficiency of high temperature hyperfiltration membrane application in the food industry. The objective of the literature survey is to compile information of value to the project. Energy and water uses in selected food process are reviewed to indicate applications for high temperature hyperfiltration membranes. Typical wastewater treatment technologies used in these food industries are briefly reviewed. The current and future effluent limitations for these food industries are discussed. The literature of membrane use in the food industry is reviewed to provide information useful in conducting the field study phases of this project.

METHODS USED IN THE LITERATURE SURVEY

The food processing information was provided by the National Food Processors Association. Typical wastewater treatment technologies were determined from the literature on industrial treatment of food processing wastewater. The information on current and future effluent limitations was collected from the Code of Federal Regulations and discussions with Environmental Protection Agency personnel.

A computerized search service was utilized in compiling the literature on membrane research. Membrane manufacturers and vendors as well as other organizations were also contacted requesting information on membrane research and applications.

CHAPTER III

PROCESS DESCRIPTION

INTRODUCTION

In 1974 the canning industry used 87×10^{12} Btu of purchased energy. About 21×10^{12} Btu were used as hot water. Even though the canning industry is distributed throughout the country, 35% of the canned fruits and vegetables are produced in California.

Energy use variation by product is illustrated in Table 1. Energy used by product ranged from 1.9×10^6 Btu per raw ton for cherries to 8.7×10^6 Btu per raw ton for peas. The style of the final product is also a large factor in energy use. For example, the range for tomatoes is from 1.0×10^6 Btu per raw ton for tomato juice up to 8.4×10^6 Btu per raw ton for tomato paste as reported in Reference (1).

The fruit and vegetable processing industry provides the market for a major portion of the nations fruits and vegetables. Approximately 90% of the beets, 80% of the tomatoes, and major fractions of the standard fruits and vegetables are preserved by this industry. Approximately 1700 plants process about 30 million tons of raw fruits and vegetables annually.

Fruit and vegetable processing plants use large quantities of water and discharge large volumes of effluent. During the highly seasonal periods of operation, the demand for water can be a very high fraction of the available potable water supply. Raw foods are cleaned and rendered wholesome for human consumption within these plants. Therefore, the food processing plant must be sanitary at all times. Large volumes of water are used to clean, transport, sanitize and cook food products.

There are variations in the waste stream characteristics since the variations of the vegetables and the climatic conditions in which they are grown are uncontrolled variables. Generally speaking, 45% of the water discharged goes directly to municipalities, while 25% is discarded to some kind of land treatment with 30% being discharged to

Table 1. Summary of canned fruits and vegetables industry processing volume, total energy use, and derived energy requirements by segment in 1973.

Segment	1973		1973		Derived Btu's Per Ton
	Raw Tons Processed		Total Energy		
	(1,000 tons)	(%)	(Bil. Btu's)	(%)	(Million)
Canned fruits	4,723	32.6	17,000	32.4	3.60
Canned vegetables, excluding tomatoes	3,634	25.0	15,600	29.7	4.29
Canned tomatoes	<u>6,150</u>	<u>42.4</u>	<u>19,900</u>	<u>37.9</u>	3.24
Total	14,507	100.0	52,500	100.0	

Source: Estimates prepared by Development Planning and Research Associates, Inc. for the Federal Energy Office/U.S. Department of Commerce, under Contract No. 14-01-0001-1652, July 1974. (Reference 1)

navigable water. Wastewater volume and organic strengths vary among the days of the operating season and the seasonal periods of operation. Treatment facilities to handle such seasonal and daily variations of wastewaters are designed to handle peak volumes of intermittent flow rather than constant flow rates of fixed composition.

PROCESS STEPS

The principal processing steps of the canning industry are listed below (not necessarily in the sequence employed in any specific plant).

<u>Stage of Process</u>	<u>Representative Steps</u>
Raw product cleaning	1. Precleaning 2. Sorting (initial grading)
Raw product preparation	3. Trimming 4. Coring and pitting 5. Cutting 6. Peeling 7. Grading and inspection 8. Transporting
Converted product handling	9. Blanching 10. Mixing and adding 11. Pulping 12. Straining 13. Cooking in vat 14. Container filling 15. Exhausting and sealing 16. Retort cooking (thermo-processing) 17. Container cooling 18. Packing (labeling, casing) 19. Warehousing-storage 20. Plant cleaning

WATER USE

In current practice in the handling of fruits and vegetables, the first large quantity of water used is in the washing of the product. The purpose of the washing and rinsing are: 1) removal of soil, dust, pesticides, microbial contamination, insects, and their residues, 2) removal of adhering juices, products of respiration or of spoilage, 3) the removal of extraneous matter, such as leaves, stems, dirt, etc., 4) removal of solubles or insolubles, such as occur during cutting,

coring, peeling and blanching, 5) the extraction of solubles such as preservative salts or acids. The quantity of water used in washing and rinsing operations may be as much as 50% of the total. Commonly water that has previously been used as cooling water, is used in the washing of the raw products. Detergents may be used to wash vegetables, particularly those grown in contact with the soil or those harvested by a mechanical device. Hot water and steam blanching serve to promote cleanliness of vegetables subject to this treatment.

Water has also been used extensively in conveying products within the plant. One advantage to this method of conveyance has been the sanitary nature of its use. Significant disadvantages of water conveying are the leeching of the solubles from the product such as sugar and acids from the fruit, and sugar and starch from corn, beets and carrots. Consequently, the ultimate disposal of water used in transport will involve the handling of sugars, starches, and acids.

Peeling of fruits and vegetables is another process which commonly involves the use of large amounts of water. Water may be used as the source of energy for puffing and loosening the peel, or may be used as a pressure spray to remove the peels that have been loosened chemically or thermally. Hot caustic solutions are widely used in strengths ranging from one percent to as high as 18 percent. The peeling solution is recirculated until it becomes contaminated. The residual caustic soda is thoroughly rinsed from the surface of the peeled fruits and vegetables.

Blanching of vegetables is done for a variety of reasons. Blanching can be accomplished by water at 150 to 210°F range or by steam at approximately atmospheric conditions. The pollution load from blanching is a significant proportion of the total pollution load in the effluent streams from the processing of certain vegetables.

The sanitary code in most states requires that cans be washed before filling. Normally the cans are flushed with a relatively large volume of water under high pressure as they travel over a conveyer in an inverted position. In this context, cans and glass are considered as "containers". If the product to be placed in the container is

a hot or heated product, it is usually necessary or desirable to have the glass container preheated during the washing process. The water used in washing is normally quite hot and must be clean and sterile.

The final major water use in the operation is the clean up. Normally the plant and equipment are cleaned at the end of each shift, usually by washing down the equipment and floors with water. In some plants, it is desirable to maintain a continuous cleaning policy so that the end of the shift clean up is minimal. In some operations, clean water is used to flush out the entire system at the end of each shift to remove any residues that may harbor bacteriological growth. The wash down may be done by either water alone or water mixed with detergents and other chemicals.

ENERGY USE

The five top energy use categories within the food industry and their projected 1980 baseline energy use are (Reference (1))

<u>Rank</u>	<u>Category</u>	<u>Energy Use (Btu)</u>
1	Beet Sugar	106×10^{12}
2	Wet Corn Milling	87×10^{12}
3	Meat Packing	97×10^{12}
4	Malt Beverages	61×10^{12}
5	Canned Fruits and Vegetables	61×10^{12}

Approximately 70 to 80% of the energy in each category is distributed from boilers as steam and is used either to heat water or supply evaporators for concentrating. Evaporation involves water only indirectly. That is, water is not consumed in the process, but the removal of water is the energy consumer in the process. Many types of evaporators are used in the industry. Natural circulation, or a thermal siphon, are in use as well as force convection systems. The heating medium may range up to 365°F steam. The concentration by evaporation is effective but is energy intensive. There may also be a loss of light-weight volatile organic molecules which tend to impart aroma and flavor to the food product.

The canned fruits and vegetables category is of primary interest to the present project. Representative process diagrams are shown in Figures

Figure 1. Process Flow and Heat Input for Producing 79 Tons of Canned Spinach from 81 Tons of Raw Spinach (from Reference 2).

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Figure 3. Process Flow and Heat Input for Producing 316 Tons of Canned Tomato Juice from 336 Tons of Raw Tomatoes (from Reference 2).

Figure 4. Process Flow and Heat Input for Producing Tomato Paste From 386 Tons of Tomatoes (Reference 2).

Figure 5. Process Flow and Heat Input for Producing 207 Tons of Canned, Peeled Tomatoes from 220 Tons of Raw Tomatoes (from Reference 2).

1 through 5 (2)). The detailed energy use patterns of individual unit processes are only now being compiled. The studies reported in Reference (2) involved detailed measurements of actual production experience. Energy uses for similar processes vary throughout the industry.

As indicated in Figures 1 through 5, much of the energy is associated with hot water. In particular, retort cooking is identified as a major user of energy. Blanching, lye peeling and hot break heating are other energy intensive process steps. In the preparation of tomato paste the use of evaporators to concentrate the tomato juice were a major energy requirement, even though multiple-effect equipment was in use at the plant studied. Principal uses of hot water are:

- 1) container washing, 2) plant and equipment cleanup, 3) blanching,
- 4) pasteurizing, 5) cooking and 6) sterilizing.

CHAPTER IV

WASTEWATER TREATMENT TECHNOLOGY AND DISCHARGE STANDARDS

WASTEWATER TREATMENT TECHNOLOGY

Table 2 summarizes typical wastewater characteristics from processing many fruits and vegetables as well as specialties food products. The sources did not indicate if the values were for raw or screened waste. The tables in Table 1 are given in unit loadings such as thousand gallons (or pounds) per ton of raw product. The ranges between the minimum and maximum values are large. Many factors cause changes in waste strength, such as raw-product condition, product conveying system, process methods, cleanup methods, batch dump frequency, duration of shutdowns, type and condition of equipment and people (Reference (3)).

There are three basic treatment options used in treating fruits and vegetables and specialties food processing wastewater: 1) pretreatment prior to discharge to a city sewer, 2) full treatment prior to discharge to a stream, and 3) discharge to land.

Pretreatment of wastewater is required to meet municipal ordinances when discharged to city sewer. Pretreatment is most often by screening, however, treatment may include neutralization, flow equalization, and soil removal. Screening removes discrete waste solids, e.g., trimmings, rejects, pits, etc. Neutralization is sometimes necessary to assure the pH of the wastewater is within acceptable limits. Flow equalization controls surges of wastewater. Soil removal from the wastewater is sometimes necessary when processing root crops, e.g., potatoes, carrots, and beets. Settling lagoons, clarifiers, grit removal cyclones and fin screens have been used to remove grit.

Discharge of wastewaters to public waters (streams and lakes) requires more treatment than pretreatment. The treatment processes used are divided into three broad categories: 1) primary, 2) secondary, and 3) tertiary. Primary treatment removes a portion of the suspended

TABLE 2. TYPICAL RAW WASTEWATER CHARACTERISTICS FOR CANNED AND PRESERVED FRUITS AND VEGETABLES (Reference 3)

Crop	Flow			BOD			TSS		
	1,000 gal/ton			lb/ton			lb/ton		
	Min.	Mean	Max.	Min.	Mean	Max.	Min.	Mean.	Min.
Apples	0.2	2.4	13	3.9	18	44	0.4	4.5	21
Apricots	2.5	5.6	14	18	40	80	5	9.9	19
Asparagus	1.9	8.5	29	0.9	4.9	22	4.3	7.5	13
Dry beans	2.5	8.8	33	15	60	182	2.6	43	99
Lima beans	2.4	7.7	22	9.3	48	175	4.6	39	231
Snap beans	1.3	4.2	11.2	1.6	15	81	0.8	6.1	46
Beets	0.3	2.7	6.7	28	53	127	7.3	22	64
Broccoli	4.1	9.2	21	5.8	20	61	4.6	17	61
Brussels sprouts	5.7	8.2	12	4.2	7.5	14	2.9	15	79
Berries	1.8	3.5	9.1	11	19	40	1.4	7.1	22
Carrots	1.2	3.3	7.1	17	30	53	4.5	17	53
Cauliflower	12	17	24	5.5	16	36	2.8	7.8	22
Cherries	1.2	3.9	14	21	38	78	1.0	2.0	3.8
Citrus	0.3	3.0	9.3	0.9	9.6	26	0.7	3.7	14
Corn	0.4	1.8	7.6	12	27	64	3.6	10	27
Grapes	0.6	1.5	5.1	6.4	9.0	13	1.5	1.7	2.0
Mushrooms	1.8	7.8	28	7.7	15	28	5.1	7.3	12
Olives	-	8.1	-	-	27	-	-	27	-
Onions	2.5	5.5	10	57	57	58	5.3	17	55
Peaches	1.4	3.0	6.3	17	35	70	3.4	8.6	21
Pears	1.6	3.6	7.7	19	50	126	3.6	12	33
Peas	1.9	5.4	14	16	38	87	3.8	11	38
Peppers	0.9	4.6	16	5	32	50	1	58	170
Pickles	1.4	3.5	11	26	42	75	3.0	8.2	23
Pimentos	5.8	6.9	8.2	39	55	76	7.1	5.8	8.1
Pineapples	2.6	2.7	3.8	13	25	45	5.2	9.1	17
Plums	0.6	2.3	8.7	6.5	10	14	0.6	2.1	4.3
Potato Chips	1.2	1.6	2.2	17	25	38	22	32	48
Potatoes, sweet	0.4	2.2	9.7	39	93	217	40	57	117
Potatoes, white	1.9	3.6	6.6	42	84	167	39	128	423
Pumpkin	0.4	2.9	11	9.2	32	87	2	67	12
Sauerkraut	0.5	0.9	3.0	4.6	5.6	15	-	1.0	2.6
Spinach	3.2	8.8	23	5.7	14	31	1.8	6.1	21
Squash	1.1	6.0	22	-	20	-	-	14	-
Tomatoes, peeled	1.3	2.2	3.7	6.3	9.3	14	5.8	12	26
Tomatoes, product	1.1	1.6	2.4	2.2	4.7	9.7	5.6	10	19
Turnips	2.4	7.3	18	-	-	-	-	-	-

Note: These figures represent two different types of samples: screened and unscreened. This increases the range of values shown.

solids in the wastewater; secondary treatment removes a portion of the dissolved solids and suspended solids material; and tertiary treatment removes additional amounts of these constituents as well as other constituents not removed by the secondary treatment. Technologies listed by the Environmental Protection Agency for each category of treatment are listed in Table 3.

Land disposal is the application of wastewater onto land. Treatment is provided by natural processes (physical, biological, and chemical) as the effluent moves through the cover crop and soil mantle. Part of the water is lost to the atmosphere through evapo-transpiration, part to surface water by overland flow, and the remainder percolates to the ground water. Land treatment is divided into four processes: slow rate, also known as crop irrigation; high rate irrigation, infiltration-percolation and overland flow. The most common land treatment method used by fruit and vegetable processors is some kind of high rate irrigation by spray nozzles. According to Nemerow (Reference (4)) the treatment processes used in treating fruit and vegetable wastewaters are: screening, chemical precipitation, lagoons, spray irrigation and secondary treatment (biological oxidation).

DISCHARGE STANDARDS

The effluent guidelines and standards for the food processing industries segment being studied are covered in the Federal Register in Part 407: canned and preserved fruits and vegetable processing point source category. Part 407 is subdivided into eight subcategories:

Subpart A - Apple Juice subcategory

Subpart B - Apple Products subcategory

Subpart C - Citrus Products subcategory

Subpart D - Frozen Potato Products subcategory

Subpart E - Dehydrated Potato Products subcategory

Subpart F - Canned and Preserved Fruits subcategory

Subpart G - Canned and Preserved Vegetables subcategory

Subpart H - Canned Miscellaneous Specialities subcategory

The best practicable control technology currently available (BPT)

TABLE 3. TREATMENT UNIT PROCESSES FOR FRUIT AND VEGETABLE PROCESSORS
(Reference 3)

Primary Treatment	Secondary Treatment	Tertiary Treatment
Grit Removal Silt Removal Plain Sedimentation	Stabilization ponds	Chemical Clarification
Dissolved Air Flotation	Aerated lagoons	Filtration (mixed bed of sand)
Chemical Treatment	Activated sludge	
	Anaerobic systems Anaerobic ponds Anaerobic contact process Anaerobic filters	Reverse Osmosis
	ABF/activated sludge	
	Trickling filters	Carbon Adsorption
	Rotating biological contactors	Ion exchange

discharge limits are presently the effluent standards being enforced. Table 4 lists the allowable mass emissions of BOD₅ and TSS for fruit and vegetable processing wastewaters. The effluent pH limit is 6.0 to 9.0 for all fruit and vegetable processing wastewaters. An additional oil and grease limitation of 20 mg/l has been set for wastewaters from the following products.

- Added ingredients
- Baby foods
- Chips (potato, corn, tortilla)
- Ethnic foods
- Jams/jellies
- Mayonnaise and dressing
- Soups
- Tomato-starch-cheese canned specialities

The best available technology economically achievable (BATEA) effluent limitations have been withdrawn and will be replaced with best conventional pollution control technology (BCT) effluent limitations. The BCT for apple juice and apple projects subcategories have been established. The BCT effluent limitations for the remaining fruit and vegetable category will probably be published in the Federal Register in late 1980 (Reference (6)).

TABLE 4. FEDERAL EFFLUENT LIMITATIONS GUIDELINES FOR FRUIT AND VEGETABLE PROCESSORS (Reference 5)
(pounds allowed per ton raw material)

Commodity	Daily Max		BPT 30-day avg		Annual avg	
	BOD	TSS	BOD	TSS	BOD	TSS
Added ingredients ^a	1.90	0.00	1.10	0.00	0.72	0.00
Apple juice	1.20	1.60	0.60	0.80	--	--
Apple products (except juice)	2.20	2.80	1.10	1.40	--	--
Apricots ^b	6.00	10.72	3.62	7.48	2.52	4.66
Asparagus ^b	--	--	--	--	--	--
Baby food ^a	2.46	4.46	1.46	3.10	1.02	1.90
Beets	2.02	3.76	1.42	2.94	1.14	2.24
Broccoli	7.66	13.56	4.42	9.14	2.94	5.30
Brussels sprouts ^b	--	--	--	--	--	--
Caneberries	1.54	2.76	0.92	1.90	0.64	1.16
Carrots	3.52	6.38	2.22	4.60	1.64	3.08
Cauliflower ^{a,b}	--	--	--	--	--	--
Cherries (sweet)	2.24	4.02	1.38	2.86	0.98	1.84
Cherries (sour)	3.54	6.40	2.22	4.60	1.62	3.04
Cherries (brined)	5.74	10.36	3.56	7.36	2.56	4.76
Chips (potato) ^a	6.92	12.50	4.34	8.98	3.16	3.94
Chips (corn) ^a	3.16	5.80	2.80	4.34	1.60	3.06
Chips (tortilla) ^a	4.82	8.68	3.00	6.22	2.18	4.08
Citrus	1.60	2.40	0.80	1.70	--	--
Corn (canned)	1.42	2.64	0.96	2.00	0.76	1.46
Corn (frozen)	2.90	6.26	1.68	4.60	1.12	3.14
Cranberries	3.42	6.12	2.06	4.28	1.46	2.68
Dehydrated onion/garlic	4.90	8.86	2.92	6.04	1.96	3.52
Dehydrated vegetables	5.96	10.60	3.52	7.30	2.42	4.42
Dried fruit	3.72	6.68	2.26	4.68	1.60	2.96
Dry beans	5.00	8.96	3.02	6.26	2.14	3.94
Ethnic foods ^a	4.78	8.46	2.82	5.82	1.92	3.46
Grape juice (canning)	2.20	3.98	1.38	2.88	1.02	1.92
Grape juice (pressing)	0.44	0.80	0.28	0.58	0.20	0.36
Jams/jellies	0.84	1.52	0.52	1.08	0.38	0.72
Lima beans	7.36	13.12	4.38	9.06	3.02	5.52
Mayonnaise and dressings ^a	0.74	1.34	0.48	0.98	0.34	0.66
Mushrooms	6.02	10.72	3.56	7.36	2.44	4.44
Olives	10.88	19.58	6.68	13.84	4.78	8.88
Onions (canned)	6.18	11.02	3.66	7.56	2.50	4.56

TABLE 4. (Continued)

Commodity	Daily max		BPT 30-day avg		Annual avg	
	BOD	TSS	BOD	TSS	BOD	TSS
Peaches	3.02	5.44	1.86	3.86	1.34	2.52
Pears	3.54	6.42	2.24	4.64	1.66	3.10
Peas	4.84	8.72	3.00	6.22	2.16	4.04
Pickles (fresh pack)	2.44	4.38	1.50	3.08	1.06	1.98
Pickles (process pack)	2.90	5.26	1.84	3.82	1.36	2.56
Pickles (salt stations)	0.36	0.66	0.24	0.50	0.18	0.36
Pimentos	--	--	--	--	--	--
Pineapples	4.26	7.70	2.66	5.52	1.92	3.62
Plums	1.38	2.48	0.84	1.74	0.58	1.08
Raisins	0.86	1.56	0.56	1.14	0.42	0.78
Sauerkraut (canning)	1.00	1.78	0.60	1.26	0.42	0.80
Sauerkraut (cutting)	0.16	0.28	0.10	0.22	0.08	1.16
Snap beans	3.02	5.34	1.74	3.60	1.16	2.08
Soups ^c	8.28	14.76	4.92	10.18	3.38	6.20
Spinach	4.74	8.38	2.72	5.62	1.82	3.28
Squash	1.80	3.28	1.18	2.46	0.92	1.74
Strawberries	3.56	6.38	2.12	4.40	1.48	2.70
Sweet and white potatoes	1.80	3.38	1.32	2.74	1.10	2.18
Tomatoes	2.42	4.30	1.42	2.96	0.98	1.80
Tomato-starch-cheese specialities ^a	2.74	6.62	2.16	4.46	1.44	2.60

^aLb/ton final product^bGuidelines have not been established as of July, 1978.^cLb/ton raw ingredients

CHAPTER V

MEMBRANE RESEARCH AND DEVELOPMENT

INTRODUCTION

A literature search was made and membrane vendors were contacted to compile relevant information for this project. Information was sought for hyperfiltration (HF), i.e., reverse osmosis (RO), and ultrafiltration (UF) research and application in food processing. The search service generated 232 titles from four data bases shown in Appendix A. The membrane vendors and other organizations contacted are listed in Appendix B.

PROCESS DESCRIPTION

Hyperfiltration and ultrafiltration are pressure-driven separation processes and may be classified by the solute separated. A simplified view of the range of filtration from conventional to "hyper" is illustrated in Figure 6.

Hyperfiltration is the membrane process that is of major interest to this project. There are several configurations of HF equipment and commercially available. The major characteristics of these available systems are presented in Table 5. The dynamic HF membrane that is being studied in this project has a major difference from the other HF membranes. The dynamic HF membrane is capable of processing high temperature ($>90^{\circ}\text{C}$) liquids at high pressures and is not plugged by suspended particles.

HISTORY

The first practical HF membrane was developed by Dr. Sourirajan in 1958 at U.C.L.A. (Reference (8)). The initial objective of HF was desalination of sea water. It has been recognized that dissolved organics as well as dissolved salts can be removed by membranes. HF was proposed for use in the food processing industry by Morgan et.al., (Ref. (9)) for concentrating and purifying various foods and food process effluents.

Table 5. Comparison of Hyperfiltration Module Configurations

Membrane Material	Cellulose acetate	Poly(ether)amide	Cellulose acetate	Polyamide	Hydrous Zr(IV) oxide-polyacrylate
Configuration	Spiral	Spiral	Tubular	Hollow Fiber	Tubular
Method of Membrane Replacement	Module (on-site)	Module (on-site)	Tubes (on-site)	Module (on-site)	Chemically (in-situ)
High Pressure Limitation	Membrane Compaction	Membrane Compaction	Membrane Compaction	Fiber Collapse	None
Prefiltration Requirement	1 micron	1 micron	40 mesh (250 micron)	1 micron	40 mesh (250 micron)
Permissible Feed Range, pH	5.5-7.5	3-10	5.5-7.5	2-10	4-10
Maximum Test Temperature °C	38	55	38	38	>105

Reference (7)

The application areas suggested are listed in Table 6. Since that time, many researchers have conducted experiments with HF and UF for potential application in the food processing industry.

In 1970 (Ref. 10) the first laboratory tests were run with dynamic HF membranes treating textile wastewater. These successful laboratory experiments led to a series of textile industry research projects partially funded by the Environmental Protection Agency and addressed the recycle of both the purified water (permeate) and also the concentrated chemicals. In many cases energy conservation was also possible by direct recycle of the hot process water from textile preparation, dyeing and finishing operations. Currently a full scale 50 gallon per minute ($280 \text{ m}^3/\text{day}$) demonstration of this application is being built at La France Industries located in La France, South Carolina.

SURVEY RESULTS

The majority of HF and UF research has been conducted in the dairy industry, particularly with whey. Fruit juice, sugar, cotton seed whey and soy whey are liquids that have had considerable testing with bench-scale HF and UF membranes. Some other liquid foods or liquids related to food processing have been investigated. All the research found in this search was conducted at relatively low temperatures due to the temperature limit on the cellulose acetate and polyamide membranes evaluated ($>38^\circ\text{C}$).

Some HF membrane research has been conducted in other industries using high temperature wastewater. Brandon (1970) evaluated a dynamic HF system with wastewater from a textile dyeing and finishing operation. The feed temperature was 82°C and the permeate and concentrate were both reused in the dyeing and finishing operation. A study (Ref. 11, 12) concluded that the dynamic HF membrane was capable of processing shower water at temperatures in excess of 74°C to produce sterile permeate suitable for recycle in a manned space mission.

Several membrane vendors are conducting pilot-scale studies in the canned fruit and vegetable industry, but do not wish to make the details public at this time. One membrane vendor has some membrane

MOLECULAR RANGE VS. FLUX FOR FILTRATION PROCESSES

CARRE DWG. 65

Figure 5. Types of Filtration Processes by Permeability and Particle Size Separated.

Table 6. Applications of Reverse Osmosis in the Food Industry

Permeate is a Major Interest

Recovery of water from waste streams
Recovery of brines (pickles, olives, etc.) by passing salts, acids,
and water through membrane
Permeating food-grade lactose from solutions of whey protein and
lactose

Concentrate is a Major Interest

Concentrating fruit juices, coffee, and maple sap without phase
change, heat damage, or loss of volatiles
Concentrating protein, egg white, whey protein, and gelatine without
heat or shear damage to functional properties
Concentrating pectin solutions without degrading the chain length
(and hence sugar-supporting power) of the pectin molecules
Concentrating egg white, pineapple mill juice, the sugar solutions
without browning reactions
De-ashing beet sugar solutions by passing salts and water through
the membrane
Desugaring egg white by passing glucose through the membrane
Purifying whey protein and lactose by passing lactic acid and salts

applications in removing fish solubles from seafood processing industry wastewater. No membrane vendors that were contacted had any membrane applications in the specialties food processing industry. The majority of the membrane applications by the vendors in the food industry, excluding purifying process water, has been with cheese whey.

In contacting organizations with some possible membrane experience, it was found that a literature and industry survey presently being conducted by the Department of Interior, Office of Water Research and Technology, is analyzing the food processing industry for potentials for water reuse and recycling. Potential applications for HF and UF membranes are being addressed in this study. A report is due for completion in late 1980.

SURVEY ARTICLES

Sammon (Reference 13) in 1969 projected a number of possible applications for membrane separation in the food industry:

1. Concentration for fruit juices
2. Concentration for maple syrup
3. Refining of sugar
4. Instant tea and coffee manufacture
5. Beer concentration
6. Dewatering of whey
7. Desalting of colloids
8. Treatment of wastes

A more recent survey by Timmons (Reference (14)) listed potential separately for ultrafiltration and hyperfiltration:

Ultrafiltration -

- Concentration of bean extracts
- Concentration of protein in fish and meat waste streams
- Egg white dewatering
- Purification of vegetable extracts
- Concentration and dewatering of gelatins

Hyperfiltration -

- Juice and beverage concentration
- Concentration of dilute sugar streams
- Concentration of brewery waste
- Concentration of grain process waste
- Production of purified process water

An article in Canner Packer (Reference (15)) suggests the

similarity of the food process separation requirements with those of the paper industry and notes the activity at the Institute of Paper Chemistry as being technology development appropriate for the food processing industry.

Pure Process Water

Two examples have been reported which typify the use of hyperfiltration water for process improvement. Jarrett (Reference (16)) relates that the variation in mineral content for domestic water is substantial. The widespread use of monocalcium phosphate as a buffer and process-regulating additive is noted. It is well known that the optimum amount of additive depends on the mineral content of the water. Thus the time-to-time and place-to-place variations in mineral content probably cause bakers difficulty in quality assurance. He predicts that the use of hyperfiltration would result in a more uniform dough formulation, better fermentation control, and elimination of other variables.

An European bottling firm was plagued by throughput of organics even after a conventional filtration unit (Reference (17)). On occasion water had to be transported to the plant. Adoption of hyperfiltration for process water corrected the problem. The fact that only 25% of the water passed to permeate was acknowledged to be a concern. A higher recovery membrane system is needed for water conservation. It was recorded that in the workshop meeting there developed a controversy over membrane cleaning indicating that several attendees also had operating hyperfiltration systems. This controversy points out that no satisfactory universal cleaning solution exists.

Soy and Protein

Timmons (Reference (14)) presents data for separation efficiency and rate of production for membrane type membranes. The fluxes vary from 30 GFD at 1% solids to about 8 GFD at 10% solids. The separation efficiency is mostly about 90% for 1% solids concentrations or greater. Baker (Reference (18) (19)) et.al. also reports data obtained for separation of soy whey solids and the computer design of an optimal system. In both cases the soy whey originates as a high BOD stream

normally wasted. After concentration of the solids by membranes, evaporation is used to produce a useful dry concentrate. A high BOD reduction is thus achieved in the fluid stream. Timmons recommends tubular membranes for soy and protein concentration because of:

1. Ease of cleaning (feed and permeate channels)
2. Simple membrane replacement
3. Tolerance for suspended solids
4. Freedom from plugging
5. Suitability for turbulent flow

Baker points out that the soy whey has very little suspended solids and he thus used spiral wound membranes. Most of Timmons criteria are economic but the presence of suspended solids is often a compelling reason to use tubular systems.

Lawhon (Reference (20), (21), (22)) has used both ultrafiltration and hyperfiltration membranes for processing whey protein from cotton seed operations. He shows substantial reduction in COD and solids in UF permeate and further reduction in HF permeate. By judicious choice of membrane the ash content can be reduced while retaining organic (protein). He also projects the acceptability of HF permeate for on-site reuse as effluent process water thus reducing the requirement for raw water.

Concentration of Juices

Attempts at concentration of juices, i.e., apple and orange, were first reported in 1968 (Reference (23)). It was noted that aroma substances in orange juice were concentrated even by membranes of relatively low retention. However, apple juice aroma compounds were only retained by the highest retention membranes. Difficulties due to the pulp and due to operation at the high pressures required to overcome the osmotic pressure were acknowledged.

Leightell (Reference (24)) elaborated more fully that the primary contributors to the ~500 psi osmotic pressure for apple juice were the sugars and to a lesser extent, the organic acids, while the aroma compounds were present in trace amounts. These compounds are comprised of $C_2 - C_6$ alcohols, $C_4 - C_8$ esters, and $C_2 - C_6$ aldehydes for apples with

ethyl-2-methylbutyrate, hexanal and 2-hexanol giving the "apple" flavor. His experiments showed some loss of aroma, but the loss was not deemed serious. However, the 1500-psi operating conditions were too stringent for sustained operation. For orange juice, the major aroma compounds are contained in an oil emulsified phase and are readily separable. Where evaporation loses the flavor compounds by vaporization, HF retains them. He found that high pressures are required for orange juice, also. Frequent membrane cleaning or prefiltration of the feed stream is required.

Matsuura and Sourirajan (Reference (25), (26)) in a series of articles more or less summarized in a survey (Reference (27)) trace further activities by their group, some of which is based on a predictive theory. Much of their conclusions are based on an alcohol-sugar-water system for the prediction of retention of aroma. They present data (Reference (25), (26)) for osmotic pressure of several juices (apple, pineapple, orange, grape fruit, grape, tomato, tomato-salted, lime, lemon, prune, and carrot). These data were derived by HF experiments at two pressures and the use of empirical extrapolation to obtain the zero flow point at which osmotic equilibrium would occur. The results are shown in Figure 7. The osmotic pressure versus solids content relationship is nearly universal except for tomato juice. The authors also show the measured effect of changes in the fluid properties on the overall separation. It is well known that when the rejected solute diffuses more slowly away from the membrane (because of higher molecular weight) it reaches a higher concentration near the surface (concentration polarization) and thereby is passed through the membrane in a proportionately greater amount. In addition, when pectin is present the fluid viscosity is altered causing greater concentration polarization and higher passage of solute through the membrane. This effect of pectin has been noticed and studied by other authors. The diffusive effect is only slightly dependent on concentration and is most severe in tomato juice, less so in lemon or lime, and least in carrot and prune juices.

Matsuura (Reference (25)) initially recommended that two membrane stages be employed since flavor compounds were incompletely retained by the cellulose acetate membranes and since flavor compounds are retained more efficiently when treated alone in the absence of sugars. The first stage would be operated at conditions designed to separate the sugars fully while the second would be operated to retain aroma (flavor). This scenario is more attractive when the dependence of flavor retention on temperature is considered. Figure 8 shows the results for an alcohol-water separation. The alcohol (representative of flavor compounds) is retained over twice as well with temperatures of 7.5 C as with 25 C. However, the membrane size at 7.5 C is much larger than that at 25 C. So, the sugar could be separated in a (relatively) small unit at 25 C or higher and the aroma compounds could be processed in a larger unit at 7.5 C. The authors did not address any problems of extended operation at high pressure.

Matsuura (Reference (26)) in a later publication chose to evaluate the aromatic polyamide membrane for retention of the polar organic solutes comprising apple flavor. He uses theory to predict 12 specific flavor compounds as rejected better than 70% accounting for diffusive effects in the fluid. The level of retentivity is superior to cellulose acetate, but still depends on operating conditions. Low temperatures are still favored as are high operating pressures as indicated in Figure 9. The recommended second stage system is a polyamide membrane while the first stage concentration should be cellulose acetate.

Pereira (Reference (28)) extended the above work using the Sourirajan techniques to calculate separation of D-glucose, D-fructose, sucrose, maltose, and lactose by cellulose acetate. He notes the anticipated effect of pectins to reduce separation because of the increase in viscosity. Reported experiments show that CaCl_2 rejections differ widely from predictions. Pereira concludes that CaCl_2 does not exist in the feed solution as an independent entity.

Watanabe (Reference (29)) has noted the severe flux decline experienced by others in orange juice concentration. He analyzed the

Figure 7. Osmotic Pressure versus Solids Content for Juices.

Figure 8. Concentration Ratio of Aromatic Compounds versus Volumetric Recovery for Cellulose Acetate Membranes at Various Operating Conditions (Reference 25).

Figure 9. Concentration Ratio of Aromatic Compounds versus Volumetric Recovery for Polyamide and Cellulose Acetate Membranes at Various Operating Conditions (Reference 25 and 26).

deposits to consist of pectins and an insoluble component similar to cellulose. He also found that the ease of cleaning was related to the conditions of operation. Fouling produced at low velocity was easier to clean. The materials deposited on a high retention (rejection 97%) membrane were removed more easily than those on a lower retention (rejection 80%) membrane.

The above study continued (Reference (30)) with experiments on laboratory fluids containing pectin and cellulose. He found repeated evidence of pectin at a critical concentration (approximately 1%) in fouling situations. The hydraulic resistance of the fouling layer followed the curve shown in Figure 10 for low and high rejection membranes. In some cases the layer formed was comprised of a viscous and a "film" component. The "film" layer only formed at high flux and high velocity. The membrane system studied was tubular (1.25 cm internal diameter 150 cm long); velocity of 56, 109, 165 cm/sec were run; and fluxes of 8 to 15×10^{-4} ml/cm²-sec were registered at $P = 50$ kg/cm². The film produced on the membrane is not water soluble and is much richer in pectin than the viscous layer. The authors conclude that cellulose plays no role and its presence is simply by being "trapped" in the pectin layer.

APPLICATION IN SUGAR CONCENTRATION

A cherry dyeing process produced a waste stream of 2-3% solids having sugars, dye, and other related substances. The waste was concentrated to about 25% solids and was reused directly, as was the permeate (Reference (31)). In the same report, an account was given of the water used to lubricate the cutters for candy. The sugars in the water were concentrated for reuse continuously. Water from the can washing following closure at a chocolate syrup installation was processed by HF (Reference (32)) to a reusable concentration. In each of these reports the BOD of the waste stream was reduced by the recycle of the valuable (when concentrated) solute, mostly sugar. In at least the latter case, the washing is conducted at 160°F so that if the separations could be performed at high temperature energy can also be

Figure 10. Relationships between concentration of pectin in deposit and resistance of deposit to water permeability. (Reference 30)

conserved. The concentration of maple sap has been reported (Reference (33)). Sugar concentration is an important function in the fruit juice processing already mentioned.

As will be pointed out in the following, sugar concentration using membranes will require attention to several important details. Although sugar is relatively easy to separate, a membrane must be selected which will provide high separation efficiency. Processing of sugar, as with many other food products, will require attention to flow passage geometry and velocity of flow. The concentration polarization which affects osmotic pressure will depend not upon the solution but also is highly dependent upon these design variables. Temperature affects the solution properties and general conditions are improved with higher operating temperatures as viscosity is lowered.

Merson (Reference (34)) illustrates the dominating importance of fluid-side properties and osmotic pressure by adopting a membrane model having infinite permeate rate. Thus the solution adjusts to a concentration at which the osmotic pressure at the membrane equals the driving pressure. The rate of water flux is controlled by solute diffusion according to generally accepted conservation of mass equations.

Merson presents his theoretical solution and data for the flux obtained with a 10% sucrose solution shown in Figure 11. In Figure 11, figure $\bar{J}$ represents the flux, Y the channel height of a two-dimensional, flat plate configuration, D the diffusion coefficient, $\bar{U}_0$ the flow velocity, and X the channel length. In a situation where $X = 2\text{mm}$, $\bar{U}_0 = 1\text{ m/sec}$, and $D = 8 \times 10^{-11}\text{ m}^2/\text{sec}$. $DX/\bar{U}_0 Y^2 = 2 \times 10^{-5}$ which, by extrapolation, predicts $\bar{J}^2 Y/D$ of about 100. The predicted $\bar{J}$ is then $4 \times 10^{-6}\text{ m/s}$ or about 8 GFD. This theory does not purport to predict any effect of fouling and the result shown is only valid for an operating pressure of 1000 psi, 10% solids.

Randall (Reference (35)) has evaluated membranes in the osmotic mode (not reverse osmosis) for concentration in sugar beet processing. Figure 12 corresponds to one of the processes he evaluated. In the process, purified thin juice from the beets normally is concentrated by evaporation until crystals can be extracted leaving molasses. The

Figure 11. Experimental and Calculated Permeation Rates for 10% Sucrose Solution Fed at 1000 psi (Reference 34).

molasses is rediluted for final sugar extraction by salt precipitation. With the membrane unit absent, the process is standard. The function of the membrane is to transfer, by osmosis, a portion of the water from the thin syrup fluid to the molasses fluid thereby diminishing the energy at the evaporation device and the water required in the dilution step. The process calls for a membrane at 70°C but the authors, for lack of a high temperature membrane, showed proof-of-principle at lower temperature. Fluxes obtained were representative of HF experience and varied in a predicted way on the solids content difference across the membrane. The experiment was conducted under some difficulty as the molasses at low temperature is highly viscous leading to severe concentration polarization on the downstream of the membrane.

Pereira (Reference (28)) continued the work of Matsuura and Sourirajan on sugars. The work extended previous calculations and experiments showing essential corroboration and supporting the observation that rejection of sugar is independent of concentration. Further, careful study of the effect of pectin on the mass transfer in the boundary layer is relevant to juice studies but also illustrates that minor constituents in sugar concentration efforts may modify the viscous properties and hence the flux levels. As already mentioned, pectin can form insoluble films of serious effects in juice processing. In the chocolate situation (Reference (32)), cocoa butter was suspected of causing a flux decline which was of a correctable nature.

MISCELLANEOUS

Various wheys and starch wastes have been studied for combined recovery of useful material and reduction of environmental problems. Rosenau (Reference (36)) reports work on ultrafiltration to preconcentrate potato starch wastes prior to evaporation to solids. Minturn (Reference (37)) studied a variety of wastes from potato grinding, wheat starch fluid (for removal of a fine colloid), potato peeling wastes and several wheys for protein recovery. Eykamp (Reference (38)) reported large differences in laboratory and field fouling tendencies. He cites, in whey, that fat, kappa casein, shattered casein, and metabolites of bacteria have been identified as contributing factors

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Figure 12. Estimated Use of Membrane for Concentration of Sugar in Beet Processing (Reference 35).

in fouling. He notes that noncellulosic membranes are harder to clean, and further that membranes need broad chemical resistance to withstand the use of aggressive cleaning agents.

Caffeine concentration was reported (Reference (39)) by HF and UF processes. The rejection by Havens membranes (cellulose acetate) of salt rejections 38% and 92% had caffeine rejections substantially the same for both membranes. Only mild concentration and pressure effects were noted. The 92% rejection membrane unit was used to concentrate the fluid beyond 10,700 mg/l (saturation) at constant flux to 15,000 mg/l. Above 15,000 mg/l concentration, the flux declined.

Ripe olive process wash water contains colors and adverse flavors preventing its reuse. Processing with a ZrO UF membrane (Reference (37)) produced an imperfect odor rejection and insufficient color removal. Use of ZrO-PAA HF provided a very clear permeate with no odor. In both cases the membrane flux suffered an initial reduce by about 67%. Variation of pH during operation of the UF membrane caused a decline perhaps indicating the precipitation of a fine particle.

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APPENDIX A

COMPUTER LITERATURE SEARCH

(Abstracts of Literature Found Are Available As An Internal Document)

AN ONLINE LITERATURE SEARCH ON
ULTRAFILTRATION, HYPERFILTRATION AND REVERSE
OSMOSIS IN THE FOOD PROCESSING INDUSTRY

A Service for

Business
Industry
Government
Professions

from

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S-2755
11 December 1979

Table A1. Data Base for Literature Search

<u>Data Base</u>	<u>Years</u>	<u>No. Titles</u>
National Technical Information Service	1964-1979	6
Compendex	1970-1979	27
Food Science and Technology Abstracts	1969-1979	155
Food Adlibra	1974-1979	30
Pollution Abstracts	1970-1979	14

APPENDIX B

**MEMBRANE VENDORS AND OTHER ORGANIZATIONS CONTACTED
by Engineering Science Survey Letter**

MEMBRANE VENDORS

Abcor, Inc.
Ajax International Corporation
Anderson, J. W. Col., Ltd.
Aqua Media
Aqua Mex, S. A.
Bend Research, Inc.
Chromalloy American Corporation
Continental Water Conditioning Co.
Crane Company
Culligan USA
DDS Ro-Division
Desalination Systems, Inc.
DORR-Oliver, Inc.
Dow Membrane Systems
Du Pont
Fluid Systems Division
Infilco-Degremont, Inc.
Ionics, Inc.
Neptune-Microfloc, Inc.
Millipore Corporation
Mott-Brandon Corporation
OSmönics, Inc.
Permutit Company, Inc.
Polymetrics, Inc.
Rev-O-Pak, Inc.
Romicon, Inc.
Sepratech
Union Carbide Corporation
Water Services of America, Inc.
Westinghouse Electric Corporation

OTHER ORGANIZATIONS

Oak Ridge National Laboratory
Texas A & M Department of
Agriculture, Western Utiliza-
tion Research and Development,
Division of Agriculture
Research Service
U.S. Department of Commerce,
Office of Water Research
and Technology
U.S. Department of Interior,
Office of Water Research
and Technology