A Study of Some Physical and Chemical Parameters affecting Nonsugar-Sugar Partition with Cellulose Acetate Membrane

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Introduction

Water shortages in recent years have prompted the development of better recovery methods. Several publications, including Okey and Stavenger (2)[2] on waste water and Vos, Hatcher, and Merten (3) on lifetime of cellulose acetate membranes, have indicated that the solvent phase, water, can be separated from dissolved solids in solution of waste water, sewage, and seawater by utilizing the reverse osmosis process.

Studies reported in this paper consider cellulose acetate membranes of high, medium, and low flux and the effects which feed juice variables of pressure, temperature, pH, and R.D.S. have on 1.—partitioning of nonsugar from sugar, and 2.—lifetime of the membrane.

While it is not within the scope of this report to define exact process applications, it is conceivable that in the future reverse osmosis may be used to increase the purity of process streams or molasses by partitioning nonsugars from sugar and, consequently, increasing white sugar production through nonsugar and color elimination.

Theory and Discussion

Reverse osmosis can be best understood by considering the theory of osmosis. If a container is divided into two compartments by a partition of semi-permeable membrane, as shown in Figure 1, and solutions of different concentration are placed in the compartments, the solvent from the less concentrated solution will pass through the membrane until the concentration of both solutions are equal. If a pressure which is greater than the osmotic pressure is applied to the more concentrated solution, a migration of solvent from the more concentrated solution to the less concentrated will take place. Thus, we have reverse osmosis.

In theory, the purification of a process stream involves a two step application of molecular sizing. Steps 1 and 2 are applicable in the below listed order or reverse order.

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[2] Numbers in parentheses refer to literature cited.

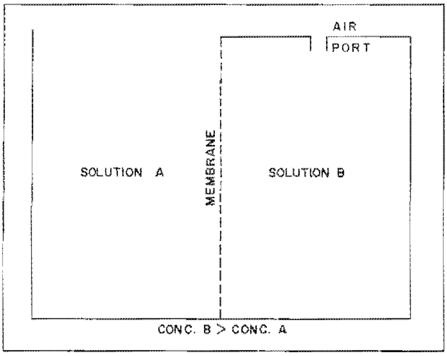

Figure 1.—A container which is divided into two compartments by a membrane - depicting the theory of osmosis and reverse osmosis.

Step 1. Choose a membrane which has the characteristic of high flux and correct microporous sizing to pass sucrose and other lower molecular weight material in the product but retains higher molecular weight material and color in the effluent.

Step 2. Process the product from Step 1. by utilizing a tighter low flux, membrane which retains sucrose in the effluent but passes lower molecular weight impurities.

Equipment and Procedures

Two of the more common reverse osmosis approaches are the plate and frame, using a membrane sheet and a plate and frame shell, and the tube type. This paper considers experiments performed with a tube type which uses a spirally wound fiberglass outside for support, a middle layer of longitudinal strands of fiberglass, and an inside layer of cellulose acetate. Some of the advantages for using the tube type are:

1. Self support.
2. Individual modules can be easily bypassed for cleaning and maintenance.
3. The juice is exposed to the entire membrane area without baffles.

The experimental modules which contained seven eight-foot lengths of ½ inch I. D. tubes and 7 sq ft of membrane area, were assembled parallel in different planes and connected in series by turn-arounds at the ends. End pieces held the turn-arounds in positions and formed a unit with a plastic shroud which covered the tubes.

As shown in Figure 2, the feed juice was transported through a series of modules by a high pressure, variable volume pump. At the outlet of the last module a spring loaded pressure regulator

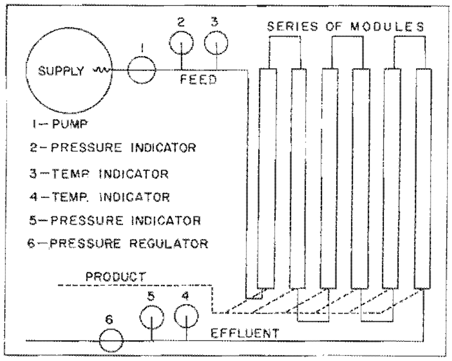

Figure 2.—Flow scheme of reverse osmosis system.

maintained a constant pressure through the system and allowed a volume of solution, the effluent, to pass out of the system. The terms feed, effluent, and product are derived from water treatment. Dissolved solids are held inside the tube as effluent while water with a low amount of dissolved solids pass through the membrane wall as product.

The analyses used for this experimental work are common to the industry and will not be discussed further.

Results

In Figure 3, results of pressure tests performed on intermediate flux modules are graphically illustrated. Module characteristics were established at 200, 400, and 600 psig pressure levels by maintaining a constant feed of dilute molasses.' Feed juice constants were: temperature 45°C, R.D.S. 11.5, pH 7.1, and apparent purity 62.0.

Graph 3 illustrates the net effect of the product contents at the three pressure levels. This results from the direct increase in product flow with increase in feed pressure, Graph 1, and the decrease in product R.D.S. with the increase in feed pressure as illustrated in Graph 2. The ratio of nonsugar to sugar in the product increases from 200 to 400 psig feed pressure but is nearly constant in the 400 to 600 psig range. By comparing Graphs 3 and 4, potassium and chloride seem to form curves similar to the nonsugar curve while sodium forms a curve similar to the sugar curve. Since the maximum amount of nonsugars

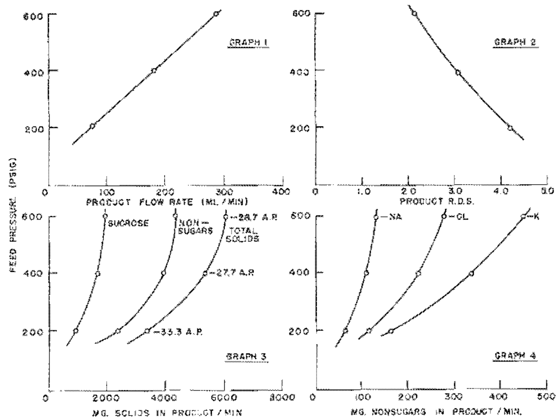

Figure 3.—Pressure characteristic of membrane - feed pressure correlation to product flow rate, product R.D.S., product solids, and product nonsugars.

were passed by the module in the 400 to 600 psig pressure range, 600 psig pressure was chosen as standard for establishing the characteristic of the module when subjected to change in feed R.D.S. and feed temperature.

The effects of changing the feed R.D.S. on the product are shown in Figure 4. Feed juices were prepared by diluting 7.1 pH, 59.7 apparent purity molasses to 29.5, 18.7, and 11.2 R.D.S. for the tests. The system temperature and pressure were maintained at 45°C and 600 psig.

Graph 7 shows the results of the increase of products R.D.S. with increase in feed R.D.S., Graph 5, and the decrease of product flow with increase in feed R.D.S. as shown in Graph 6. Sharp increases of total nonsugar and total sugar in the product were noted as the feed R.D.S. was increased from 11.2 to 18.7 R.D.S., whereas only small changes were seen as the feed R.D.S. was increased from 18.7 to 29.5. Total sugar in the product increased at a slightly faster rate than the total nonsugar as indicated by the apparent purity.

Graph 7 indicates that with a 15 R.D.S. feed the module removes nearly the maximum amount of nonsugars while sacrificing the least amount of sugar. Higher feed R.D.S. values cause loss of more sugar while removing the same amount of nonsugars.

The effects of feed temperature on the module were evaluated at 28°C, 37°C, and 45°C. Feed juice was prepared by diluting 59.8 apparent purity, 7.1 pH molasses to 10.15 R.D.S.

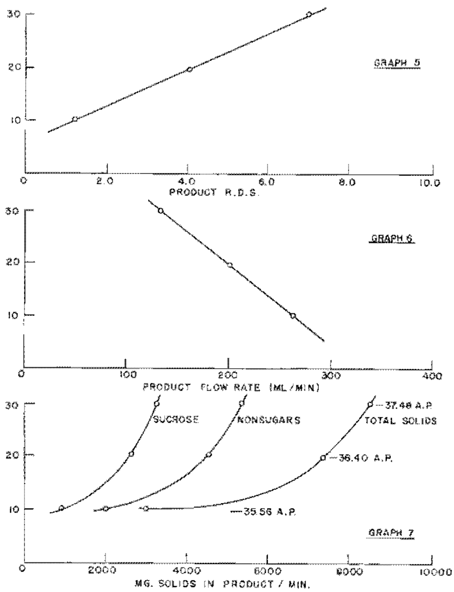

Figure 4.—Effects of feed R.D.S. on the membrane - feed R.D.S. correlation to product R.D.S., product flow rate, and product solids.

As indicated in Figure 5 - Graph 8, the flow rate increased 35 percent as the temperature increased from 28°C to 45°C. Higher temperatures were not recommended because of possible destruction of the membrane.

Graphs 9 and 10 indicate increases in total solids and R.D.S. with increase in temperature. Sugar passes through the membrane at a slightly higher rate than the nonsugars with the increase in temperature as indicated by the increase in apparent purity.

The diluted molasses tests can be best summed up by the theoretical material balance derived from experimental data as diagramed in Figure 6. This four stage system is connected in series with the effluent from the first acting as the feed for the second, etc. Product is passed by the membrane in each stage.

It is assumed that the feed has a temperature of 45°C, a pressure of 600 psig, and R.D.S., apparent purity, and flow rate values as listed in the diagram.[3] The product is assumed to have a constant apparent purity. All other information is taken from the graphs or calculated from the graph information.

Each consecutive feed has a greater R.D.S. and apparent purity and less flow than the previous feed. Consequently, each

[3] These were values used in the tests.

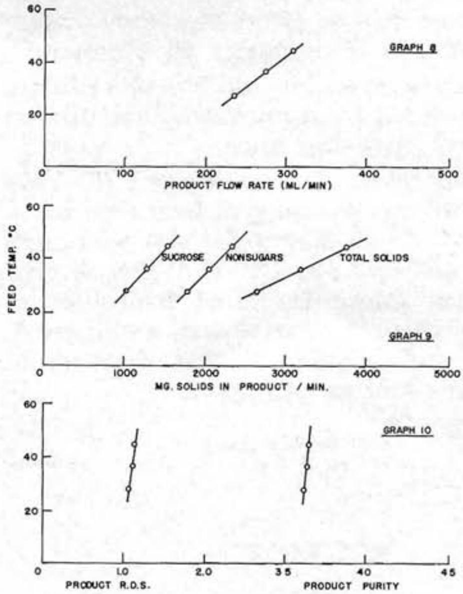

Figure 5.—Effects of feed temperature on the membrane - correlation of feed temperature to product flow rate, product solids, product R.D.S., and product purity.

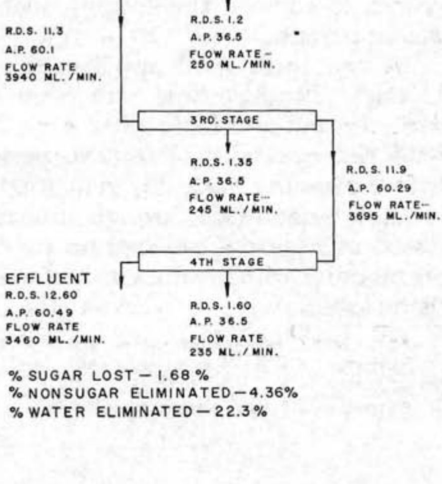

Figure 6.—A four stage system which passes smaller molecules than sucrose in the product. Effluent from each stage is the feed for the next. Note the increased quality of the each effluent as compared to the original feed.

product has a greater R.D.S. and less flow than the previous product. 1.68% of the sucrose is lost in the product, while 4.36% of the nonsugars and 22.3% of the water are eliminated.

Studies of the lifetime and irreversible fouling of cellulose acetate membranes were considered by using raw juice, diluted 10-11 R.D.S. high raw pan stock, and thin juice.

Havens Industries and Vos, Hatcher, and Merten (3) have established that cellulose acetate membranes will hydrolyze when exposed to hot alkaline solutions. An eleven day test with high flux cellulose acetate modules and feed juice, 10-11 R.D.S. diluted high raw pan stock and thin juice,[4] indicated hydrolysis of cellulose acetate at the end of this period as shown in Figure 7. The membrane became soft and pliable. A 30% decrease in product flow due to irreversible fouling was noted.

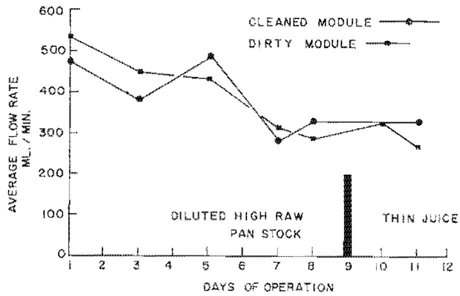

Figure 7.—Average flow rate per minute per module (7 square feet of membrane area) is plotted versus days of operation.

A polyurethane foam plug, which was pumped through the modules every 24 hours to remove the colloidal buildup on the tube wall, had no effect on the flow rate of the product. At the end of the 9th day, a 2% HCl solution was pumped through the system to remove the fouling material but no increase in flow was apparent.

A raw juice feed[5] and low flux modules were evaluated for 11 days. Product flow rate from modules which were cleaned with the polyurethane plug each 24 hours, Figure 8, showed a 50% decrease in flow for the period. Product flow rates taken before cleaning each day remained constant.

Raw juice feed[6] through intermediate flux modules, as illustrated in Figure 9, showed no product flow at the end of 4 days on modules which were cleaned every 24 hours with the polyurethane plug.

[4] pH - 8.5-8.8, temperature - 45° C, pressure 600 psig.

[5] pH - 6.0, R.D.S. - 10.5, pressure - 600 psig, temperature 35° C for 3 days and then 50° C.

[6] Pressure - 600 psig, temperature - 50° C.

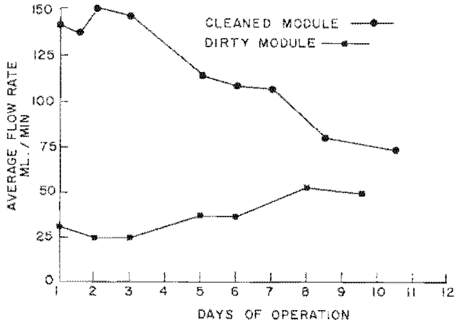

Figure 8.—Average flow rate per minute per module (7 square feet of membrane area) is plotted versus days of operation, using raw juice as feed.

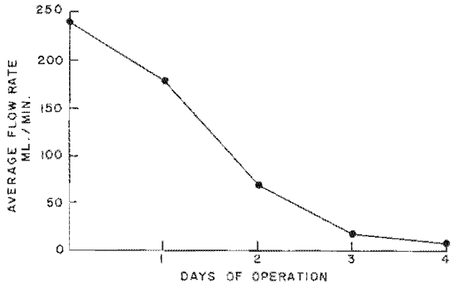

Figure 9.—Average flow rate per minute per module (7 square feet of membrane area) is plotted versus days of operation, using raw juice as feed.

Summary

Reverse osmosis is a potential means for separating sucrose from nonsugars and color. Development and testing of membranes which are not subject to permanent fouling and decomposition when in the environment of process juices must be accomplished before serious process application can be considered.

Acknowledgment

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