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Preliminary Concentration of Maple Sap Using Reverse Osmosis

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Preliminary Concentration of Maple Sap Using Reverse Osmosis

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1.0 Introduction

The traditional production of maple syrup by boiling in open pans is being re-appraised in the light of fuel and energy conservation. The dilute sap collected at between 1° to 4° Brix is concentrated to 65° Brix by boiling for an average 30 - 40 fold concentration. With reverse osmosis (R.O.) systems, high permeate flow rates are easily attained at low soluble solids levels. Preliminary maple sap concentration is thus a logical application of R.O. as a concentration increase from 2° to 4° Brix will remove 50% of the original water and yet the product will still have a low osmotic pressure.

This report describes some of the demonstration work carried out at the Kentville Research Station in the spring of 1974.

2.0 Review of Literature

Early work in the concentration of maple sap using reverse osmosis was carried out at the Eastern Utilization Research and Development Div., U.S.D.A. (Willits et al., 1967; Underwood et al., 1969). The U.S.D.A. group demonstrated the process at the laboratory and pilot scale levels.

In the production of maple syrup the heat involved in the boiling process is necessary for the development of the characteristic flavour and colour of the product. Willits (1967) points out that boiling of 40⁰ Brix (or higher) syrup favoured the development of the colour and flavour. The removal of the first 50% of the water by using the low temperature R.O. system should not affect colour and flavour development. In their studies Underwood et al. (1969) used commercial cellulose acetate spirally wound membranes produced by Gulf General Atomic. They report no loss of sugars in the permeate liquid and very little loss of organic salts.

Alwin (1971) also discusses the use of a demonstration R.O. unit in the concentration of maple sap. He notes the problem of yeast build up which decreases equipment efficiency and necessitates clean-up procedures, and the decrease in benefits as the R.O. is used for higher levels of concentration. Underwood (1969) used an in-line ultraviolet water sterilizer to minimize a build up of micro-organisms.

The above authors note the savings in energy over conventional evaporating pans. Underwood (1969) calculated a 54% fuel cost reduction based on the assumption of using #2 fuel oil and 1968 price levels. For many of the smaller production units the energy savings would not be as clear cut, as the energy for boiling the sap is often obtained from wood gathered on the farm rather than oil (Dillon, 1971). Fuel still accounts for 9% of the cost in Dillon's economic survey of maple production in Ontario. It is interesting to note that Underwood estimates fuel costs at about 50¢ per gal with oil, while Dillon gives fuel costs from 42¢ to 58¢ per gal. Both estimates were done in 1968.

3.0 Materials and Methods

3.1 Apparatus

The demonstration trials were conducted using an Osmo 3319 reverse osmosis pilot plant unit (Fig. 1) with a 33 sq ft spiral wound membrane. The membranes used were cellulose acetate (Osmo 334-89), intermediate rejection membranes which will reject 85 - 90% NaCl. The basic R.O. unit is equipped with a 3/4 hp pump capable of generating about 180 psi. Higher pressure trials were performed using a high pressure Cherry-Burrell triplex pump.

3.2 Material

Maple sap was harvested and frozen in 80 lb blocks and held in frozen storage by Oxford Frozen Foods until the trails were conducted in June. Prior to the trials the material was thawed at room temperature without additional heat. Details on the harvested sap, as to stage of the run, or holding time before freezing, were not available. The thawed sap was of relatively poor quality, as it appeared cloudy. Microscopic examination of the sediment showed a high concentration of yeast and a plate count of 1×10^4 yeast/g was found for the raw sap. Tests 1-9 were run with the sap in this condition, but for tests 11-13 the sap was first filtered through a Carlson plate filter using 5, 50 micron and 3, 25 micron filters. The filtered sap was clear.

3.3 Test Procedures

A series of seven tests were run with raw or filtered sap making one pass through the membrane module at 180 psi. Tests for one pass through the module with filtered sap were also run at 420 and 620 psi. Concentrated sap from the single pass trials was collected and used for tests on subsequent passes through the membrane. The sap was subjected to up to four passes through the module.

For the low pressure runs, the built-in pump of the Osmo was used, running in a suction mode. For the higher pressures a Cherry-Burrell pump was employed. The sap was passed through an 80 mesh stainless steel filter prior to entering the system.

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Before each run the module was flushed with fresh water. After run 10 a decreased permeation rate was observed and the membrane was thoroughly cleaned using an acid detergent.

Enery consumption of the pumps was measured during the trials with a simple induction coil ammeter.

4.0 Results and Discussion

The general results of the eighteen tests are summarized in Table 1. It can be seen that there was no measurable loss of sugars into the permeate in any of the trials. The retention of all the sugar in the concentrate is essential for effective use of R.O. in this application.

Permeation rates and the degree of water removal with each pass through the membrane were calculated for the tests conducted. The tests are summarized in groups; runs 1-5, runs 6-10, runs 11-13, run 14, run 15 and runs 16-18. The percentage removal of water for multiple pass trials was calculated on the basis of the original water content.

The concentrate collected from runs 1-3 represented 1 pass material, which was used as feed for the 2nd pass (run 4). The concentrate from run 4 was collected and used as feed for the 3rd pass (run 5). A similar sequence was used for runs 6-9 with 6 and 7 the first pass, 8 the second and 9 the third. In runs 11-14 the same sequence was followed.

As anticipated, permeation rate decreased with increased concentration of feed material (Fig. 2). Even at this low concentration the decrease in rate is appreciable. Combined with the decrease in rate due to concentration, there is a marked decrease in permeation rate with an increase in microbe numbers in the sap and the consequent plugging of the membranes. This effect is seen when runs 1-5 are compared with 6-9 in Fig. 2. While the

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module was flushed with fresh water prior to each set of runs, it can be seen that the permeation rate for the second set of runs, 6-9 (Fig. 2) had fallen off considerably. After the completion of the second set of runs, the module was thoroughly cleaned using an acid detergent and carefully flushed with fresh water. The clean-up treatment restored the permeation rates to a level comparable with the initial runs (runs 11-13, Fig. 2).

A large increase in permeation rate was obtained with the higher pressure operation. The rate of water removal at 620 psi was almost double the rate at 180 psi (runs 16-18 vs runs 11-13) for comparable feed concentration and condition. Whether the increase in flow rate is economical would depend on the relative costs of increased pump requirements at the high pressure vs additional membrane area at the lower pressure to achieve the same flow.

Table 2 gives the percent water removal from the sap on the basis of the original water content (for example, in runs 1-5, 57.3% of the water in the original sap had been removed after three passes through the system.). The levels of removal were quite consistent for the four sets of tests, with the degree of separation being very similar at 180 psi or 620 psi. The H_2^0 removal per pass is lower than the figures given by Alwin (1971). He claims to have about a 50% removal of H_2^0 with one pass through the membrane. The percentage removal of water in one pass for high microbe count sap was calculated from Alwin's data to be about 25%. This figure agrees well with the present data and is an indication of the importance of the microbial content of the raw sap.

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Microbial counts for the sap were quite high ranging from 5×10^4 to 4×10^6 micro-organisms/gram. A decrease from 1.37×10^5 to 4.7×10^3 was obtained by the filtering, but after one pass through the membrane module the count had increased to 7.8×10^4 . The increase in count following the membrane process is due both to the physical concentration of the yeast (i.e. no yeast cells pass through the membrane, thus, the yeast cells are concentrated at the same time as the sugars are concentrated) and to microbial growth in the sap and in the module. All the micro-organisms in the sap were identified as yeasts.

The necessity of obtaining good quality sap and processing it without delay is pointed out by the effect of the microflora on the permeation rates. A good sanitation and clean-up schedule for the membranes would be essential for efficient operation. A parallel system of two or more membranes which would allow for a regularly scheduled clean up and sanitation of membranes during continuous operation would be very desirable.

Energy requirements for water removal are critical to the selection of a concentration system within the limitations imposed by the necessity of boiling the syrup above 40% soluble solids to achieve proper colour and flavour. Electrical energy requirements for water removal under the various test conditions are given in Table 3. Tests 1 through 14 used only the Osmo pump and had energy consumptions from 45 to 90 watt hrs/kg of H_20 removed. The influence of membrane fouling and feed concentration on energy consumption is the same as the effect on permeation rates. An increase in energy use is seen with runs 6-9 in comparison with 1-5. The membrane cleaning reduced the energy requirement to the original level (runs 11-13). The high pressure runs of course used more energy per Kg of permeate than the low pressure as the two pumps were used. The increase in energy is seen in Table 3. The energy requirements of about 135-157 watt hrs/kg of permeate water removed at 420 and 620 psi were higher than the requirements of the low pressure system. The Cherry-Burrell pump used in the high pressure runs was larger than required so the actual energy requirements would be lower.

There are several factors which would influence the choice of system for high or low pressure operation as well as the energy use. A factor which has not been studied here is the influence of operating pressure on membrane life and permeation rate. More compaction of the membrane with the resultant drop in efficiency would be expected with the higher pressure use. Selection would depend on throughput, membrane cost, and pump costs as well as energy consumption.

Quality of syrup produced from sap initially concentrated by R.O. was the same as syrup produced by a boiling system as judged by sensory tests.

5.0 Conclusions

Removal of up to 50% of the water in maple sap can be readily accomplished using reverse osmosis. Because of the low soluble solids level in the sap, large quantities of water can be removed at a low osmotic pressure.

The permeate flow rates show the importance of a regular membrane cleaning schedule. For a continuous commercial installation one or more alternate membranes would be required to allow for clean up in the continuous system as the permeation rate drops off within hours when operating with high

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microbial count sap. The permeat flow rates given here are probably on the conservative side, as the sap used had a high microbial content. An increased separation (i.e. a greater percentage removal of water with each pass) would be expected with high quality sap. Removal of up to 50% of the water in one pass was achieved by Alwin (1971).

Studies on membrane life in this operation have not been conducted. Some figures on this factor should be available from the various manufacturers. A study on a complete seasons operation using the small pilot plant and several thousand gallons of sap should give an estimate on longer term membrane performance.

With the increased price of oil for firing a conventional evaporation system, the energy costs for the removal of about 50% of the water by R.O. looks quite attractive.

6.0 Acknowledgement

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| May | 29, | 1974 |
|------|-----|------|
| June | 4, | 1974 |

SUMMARY:

| | FEED PRODUCT | NOMINAL OP'N. PRESS | FEED % SUCR. | RUN TIME (MIN.) | CONCENTRATE | | PERMEATE | |
|-----|-----------------|---------------------------|-----------------|-----------------------|--------------|---------|----------|---------|
| RUN | | | | | VOL. L. | % SUCR. | VOL. L. | % SUCR. |
| 1 | Sap | 180 | 2.5 | 11.3 | 11.8 | 3.4 | 4.3 | 0 |
| 2 | Sap | 180 | 2.5 | 11.6 | 12. | 3.45 | 4.5 | 0 |
| 3 | Sap | 180 | 2.5 | 35.5 | 36.8 | 3.35 | 13.9 | . 0 |
| 4 | 1 Pass | 180 | 3.35 | 42.2 | 44.5 | 4.5 | 15.0 | 0 |
| 5 | 2 Pass | 180 | 4.4 | 33.8 | 34.2 | 5.6 | 10.0 | 0 |
| 6 | Sap | 180 | 2.0 | 75.0 | 66.0 | 2.95 | 26.1 | 0 |
| 7 | Sap | 180 | 2.2 | .90.0 | 72.0 | 2.85 | 27.9 | 0 |
| 8 | 1 Pass | 180 | 2.85 | 90.0 | 72.8 | 3.9 | 23.9 | 0 |
| 9 | 2 Pass | 180 | 3.9 | 71.0 | 53.2 | 5.0 | 15.5 | 0 |
| 10 | 3 Pass | 180 | 5.0 | 22.0 | 3 0.0 | 5.65 | 5.2 | 0 |
| 11 | Filtered Sap | 180 | 1.55 | 60.0 | 84.4 | 2.05 | 26.6 | .0 |
| 12 | 1 Pass | 180 | 2.05 | 47.0 | .62.3 | 2.7 | 19.9 | .0 |
| 13 | 2 Pass | 180 | 2.7 | 37.0 | 46.1 | 3.45 | 13.3 | 0 |
| 14 | Filtered Sap | 180 | 1.45 | 24.0 | 27.7 | 2.0 | 9.5 | 0 |
| 15 | Filtered Sap | 420 | 1.5 | 16.0 | 41.1 | 2.0 | 10.9 | 0 |
| 16 | Filtered Sap | 620 | 1.5 | 15.0 | 36.5 | 2.0 | 11.9 | 0 |
| 17 | 1 Pass | 620 | 1.8 | 30.0 | 64.0 | 2.45 | 23.6 | 0 |
| 18 | 2 Pass | 620 | 2.75 | 17.0 | 38.8 | 3.5 | 12.2 | 0 |

.

| Table | 2 |
|-------|---|
| | - |

| | • | • | | | | · · · | |
|-----|------|------------------|--------------------------|-------------------|-------------------|---------------------------------------|-----------------------|
| RUN | PASS | TIME (MIN.) | FEED % SOL. SOLIDS | VOL. CONC. (L) | VOL. PERM. (L) | % H ₂ 0 REMOVAL (TOTAL) | PERM. RATE (L/MIN) |
| 1-3 | 1 | 58.4 | 2.5 | 60.6 | 22.7 | 27.3 | 0.399 |
| 4 | 2 | 42.2 | 3.35 | 44.5 | 15.0 | 45.3 | 0.365 |
| 5 | 3 | 33.8 | 4.4 | 34.2 | 10.0 | 57.3 | 0.296 |
| 6-7 | 1 | 165 | 2.1 | 138.0 | 54.0 | 28.1 | 0.327 |
| 8 | 2 | 90 | 2.85 | 72.8 | 23.9 | 45.9 | 0.266 |
| 9 | 3 | 5. 71 (M) | 3.9 | 53 . 2 | 15.5 | 57.4 | 0.219 |
| 11 | 1 | 60 | 1.55 | 84.4 | 26.6 | 24.0 | 0.444 |
| 12 | 2 | 47 | 2.05 | 62.3 | 19.9 | 41.9 | 0.423 |
| 13 | 3 | 37 | 2.7 | 46.1 | 13.3 | 53.8 | 0.36 |
| 16* | 1 | 15 | 1.5 | 36.5 | 11.9 | 24.6 | 0.794 |
| 17 | 2 | 30 | 1.8 | 64.0 | 23.6 | 44.9 | 0.786 |
| 18 | 3 | 17 | 2.75 | 38.8 | 12.2 | 58.1 | 0.718 |
| | • | | | | | | , |

PERMEATION RATES AND WATER REMOVAL PERCENTAGES

*Pressure for runs 16 - 18 was 620 psi.

| | ELECTRICAL | ENERGY REQUIRE | MENTS FOR WA | TER REMOVAL |
|-------------|-----------------------|--------------------|--------------|---|
| TEST NO. | FEED CONCENTRATION | PERMEATION RATE | PRESSURE | WATT HRS/KG H ₂ 0 REMOVAL |
| | % SOLUBLE SOLIDS | L/MIN. | PSI | |
| 1-3 | 2.5 | 0.389 | 180 | 50.9 |
| 4 | 3.35 | 0.365 | 180 | 54.3 |
| 5 | 4.4 | 0.296 | 180 | 66.9 |
| 6-7 | 2.1 | 0.327 | 180 | 60.5 |
| 8 | 2.85 | 0.266 | 180 | 74.5 |
| 9 | 3.9 | 0.219 | 180 | 90.4 |
| 11 | 1.55 | 0.444 | 180 | 44.6 |
| 12 | 2.05 | 0.423 | 180 | 46.7 |
| 13 | 2.7 | 0.360 | 180 | 53.5 |
| 14 | 1.45 | 0.396 | 180 | 49.7 |
| 15 | 1.5 | 0.682 | 4 20 | 157.* |
| 16 | 1.5 | 0.794 | 620 | 1 3 5.* |

*A direct comparison using these figures is difficult as the capacity of the Cherry-Burrell pump used for the high pressure runs was much

0.786

0.718 ...

620

620

136.*

149.*

Table 3

greater than required.

1.8

2.75

17

18



Figure 1. The Osmo 3319SS reverse osmosis assembly used in the study.



