# From 8 to 35 °Brix: Research on reverse osmosis and syrup flavor and quality



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#### **Reverse Osmosis/Membrane Separation**

Increases efficiency through reductions in boiling time and fuel costs

Other effects? Chemical composition? *Flavor?* 



### Experiments

Controlled experiments conducted with commercial maple equipment at UVM PMRC Maple Processing Research Facility to investigate impacts of RO on syrup composition and flavor:

Raw sap vs. 8% Concentrate

2, 8, 12, and 15%

8% vs. 22% Concentrate



Maple Processing Research Facility (MPRF) at UVM PMRC

### **First Experiment Objective**

#### Investigate the effects of pre-concentrating sap with RO on:

# Syrup composition, properties, flavor

Does syrup produced from raw sap differ from syrup produced with the same sap concentrated by RO?





van den Berg, A.K., Perkins, T.D., Isselhardt, M.L., Godshall, M.A. and Lloyd, S.W. 2014. Effects of membrane separation on maple syrup composition and flavor. *International Sugar Journal* 116:656-665.

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Produce syrup from raw sap and *same* sap concentrated to 8%

Compare composition and flavor



#### **2 Treatments:**

#### Raw sap (2%) and same sap concentrated to 8%







Each tank fed one of two evaporators



Syrup from each evaporator collected and filtered separately

#### Packed for analysis





Repeated on 6 days during production season

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# Syrup analyses

#### Composition and properties: Color, pH, conductivity Inorganic Minerals

#### Flavor:

Volatile flavor compounds Sensory evaluation



Does attribute differ in syrup produced with raw sap and same sap concentrated with RO?

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### **Results: Syrup color**



#### **Results: Syrup properties and composition**

Parameter measured	Raw Sap (2%)	Concentrated Sap (8%)	<i>p-</i> value
Brix (°)	67.1 ± 0.2	67.2 ± 0.1	0.4142
Conductivity (µS cm <sup>-1</sup> )	171.6 ± 11.5	162.9 ± 9.1	0.1572
Light transmittance (%)	57.7 ± 4.4	45.9 ± 3.8	0.0001
рН	7.1 ± 0.05	$7.5 \pm 0.09$	0.0091
Calcium (ppm)	946 ± 77	939 ± 23	0.9164
Phosphorous (ppm)	$2.3 \pm 0.7$	4.1 ± 1.2	0.1424
Potassium (ppm)	1948 ± 37	$2009 \pm 48$	0.2694
Magnesium (ppm)	153.2 ± 7.7	133.1 ± 8.4	0.0063
Iron (ppm))	1.9 ± 0.5	$1.9 \pm 0.6$	0.9171
Manganese (ppm)	27.2 ± 5.1	$16.0 \pm 2.4$	0.1097
Boron (ppm)	1.2 ± 0.2	$0.9 \pm 0.1$	0.0625
Copper (ppm)	$0.9 \pm 0.1$	$1.0 \pm 0.1$	0.6274
Zinc (ppm)	3.2 ± 0.1	$3.6 \pm 0.2$	0.1084
Sulfur (ppm)	17.5 ± 1.2	18.7 ± 1.2	0.5443
Sucrose (%)	65.4 ± 0.9	$64.2 \pm 0.7$	0.2175
Glucose (%)	0.11 ± 0.004	$0.09 \pm 0.006$	0.0125
Fructose (%)	$0.69 \pm 0.02$	$0.67 \pm 0.02$	0.4607
Total invert sugar (%)	$0.79 \pm 0.02$	$0.75 \pm 0.02$	0.0938
Volatile flavor compounds (millions of peak area count)	$2.4 \pm 0.3$	$2.3 \pm 0.3$	0.9166

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Average composition and properties of syrup produced with raw sap and the same sap concentrated to 8%. p value for paired Student's t-test (n=6).

#### **Results: Sap, Concentrate and Permeate**

Experiment trial date	;	3/18/201	1	;	3/21/201 <sup>-</sup>	1		4/1/2011			4/4/2011			4/8/2011	
Material type	Ρ	S	С	Ρ	S	С	Ρ	S	С	Ρ	S	С	Ρ	S	С
Brix (°)	0.0	2.3	8.4	0.0	2.4	7.7	0.0	2.4	8.1	0.0	2.5	8.2	0.0	2.2	8.0
рН	6.3	7.5	7.7	5.8	6.7	7.1	6.0	7.0	7.3	6.0	7.0	7.4	6.0	6.7	7.1
Conductivity (µS cm <sup>-1</sup> )	6.3	460.7	1218.0	6.5	504.7	1217.0	3.2	398.0	987.8	3.3	456.6	1105.0	4.6	464.5	1203.0
Calcium (ppm)	0.09	49.9	196.0	0.12	60.0	198.0	0.04	41.8	141.0	0.11	50.5	173.0	0.11	52.1	193.0
Phosphorous (ppm)	bdl	0.5	3.4	bdl	0.5	2.2	bdl	0.4	1.9	bdl	0.2	1.1	bdl	0.3	1.4
Potassium (ppm)	1.1	58.8	256.0	1.0	62.8	257.0	0.5	49.9	212.0	0.5	56.1	202.0	0.7	56.7	276.0
Magnesium (ppm)	0.012	5.6	20.5	0.013	6.7	20.5	0.004	4.5	14.5	0.009	5.7	18.1	0.010	6.0	20.6
Iron (ppm)	bdl	0.1	0.2	bdl	0.1	0.2	bdl	0.0	0.1	bdl	0.1	0.1	bdl	0.1	0.2
Manganese (ppm)	bdl	5.5	22.0	bdl	5.6	18.6	bdl	4.2	14.3	bdl	5.3	17.9	bdl	6.0	21.8
Boron (ppm)	bdl	0.1	0.1	bdl	0.1	0.1	bdl	0.0	0.1	bdl	0.1	0.1	bdl	0.0	0.1
Copper (ppm)	bdl	0.1	0.2	bdl	0.1	0.2	bdl	0.0	0.2	bdl	0.0	0.2	0.03	0.0	0.1
Zinc (ppm)	bdl	0.3	1.2	bdl	0.3	1.0	bdl	0.2	0.8	bdl	0.2	1.0	bdl	0.3	1.0
Sulfur (ppm)	bdl	0.8	3.7	bdl	1.0	3.9	bdl	0.7	2.5	bdl	0.9	3.5	bdl	1.0	4.0
Sucrose (%)	nm	1.8	7.1	nm	1.8	6.1	nm	2.0	7.0	nm	1.9	6.4	nm	1.6	6.4
Glucose (%)	nm	0.07	0.24	nm	0.09	0.25	nm	0.07	0.22	nm	0.10	0.26	nm	0.09	0.23
Fructose (%)	nm	0.04	0.12	nm	0.04	0.14	nm	0.03	0.11	nm	0.04	0.13	nm	0.04	0.13
Total invert sugar (%)	nm	0.1	0.4	nm	0.1	0.4	nm	0.1	0.3	nm	0.1	0.4	nm	0.1	0.4

Composition of raw sap, 8% concentrate, and permeate for 5 experiment trials.

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#### **Results: Sap, Concentrate and Permeate**

	Permeate	Sap	Concentrate	Calculated Concentration
Brix (°)	0.0	2.4	8.1	8.1
Calcium (mg kg <sup>-1</sup> )	0.04	41.8	141.0	141.0
Phosphorous (mg kg <sup>-1</sup> )	bdl	0.4	1.9	1.3
Potassium (mg kg <sup>-1</sup> )	0.5	49.9	212.0	168.3
Magnesium (mg kg <sup>-1</sup> )	0.004	4.5	14.5	15.0
Iron (mg kg <sup>-1</sup> )	bdl	0.04	0.12	0.14
Manganese (mg kg <sup>-1</sup> )	bdl	4.2	14.3	14.2
Boron (mg kg <sup>-1</sup> )	bdl	0.04	0.09	0.14
Copper (mg kg <sup>-1</sup> )	bdl	0.04	0.15	0.14
Zinc (mg kg <sup>-1</sup> )	bdl	0.2	0.8	0.7
Sulfur (mg kg⁻¹)	bdl	0.7	2.5	2.3
Sucrose (%)	nm	2.0	7.0	6.6
Glucose (%)	nm	0.07	0.22	0.24
Fructose (%)	nm	0.03	0.11	0.10
Total invert sugar (%)	nm	0.10	0.33	0.34

Composition of raw sap, the same sap concentrated to 8%, and the calculated composition of the concentrate based on the concentration factor (3.4x)

# **Results: Syrup flavor**

Parameter measured	Raw Sap (2%)	Concentrated Sap (8%)	<i>p-</i> value
Brix (°)	67.1 ± 0.2	67.2 ± 0.1	0.4142
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Calcium (ppm)	946 ± 77	939 ± 23	0.9164
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Fructose (%)	$0.69 \pm 0.02$	$0.67 \pm 0.02$	0.4607
Total invert sugar (%)	0.79 ± 0.02	$0.75 \pm 0.02$	0.0938
Volatile flavor compounds (millions of peak area count)	$2.4 \pm 0.3$	$2.3 \pm 0.3$	0.9166

# Syrup flavor: Sensory evaluation

#### **Triangle Test:**

Is there an overall difference in flavor between 2 samples?





# Syrup flavor: Sensory evaluation



#### Triangle Tests

4 Syrup pairs 3/18, 4/2, 4/4, 4/8



Is there a difference in flavor between syrup made simultaneously from raw sap and concentrate?

# Syrup flavor: Sensory evaluation



#### Additional experiments: Higher and varied concentration levels

# Experiments to examine: 2, 8, 12, and 15% 8% vs. 22%



#### Similar methodology

Concentrate

van den Berg, A.K., Perkins, T.D., Isselhardt, M.L., Godshall, M.A. and Lloyd, S.W. 2012. Maple syrup production with sap concentrated to high levels by membrane separation: effects on syrup chemical composition and flavor. *International Sugar Journal* 114:572-576.

van den Berg, A.K., Perkins, T.D., Isselhardt, M.L., Godshall, M.A. and Lloyd, S.W. 2011. Effects of producing maple syrup from concentrated and reconstituted sap of different sugar concentrations. *International Sugar Journal* 113:35-44.



#### **Results: Syrup Color**



## **RO effects on syrup color?**



Syrup made from raw sap tends to be **lighter** than syrup made with sap concentrated to 8%

Once RO is used: Syrup made with more concentrated sap tends to be lighter than syrup made from less concentrated sap



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# **Results: Syrup composition and flavor**

#### **Composition/ Properties:** Very few (if any) significant differences

#### Flavor:

No significant differences found in sensory evaluations



#### Conclusions

#### Effects of RO on syrup composition and flavor?

# Slight to moderate effects on color

Minimal (if any) impacts on properties and chemical composition

No perceptible impacts on flavor in sensory evaluations



#### Results of all 3 experiments outlined in October 2015 *Maple Digest* article

http://www.uvm.edu/~pmrc "RO Effects on Syrup"

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Research: Reverse Osmosis

#### Effects of Sap Concentration with Reverse Osmosis on Syrup Composition and Flavor A summary of experiments conducted at the University of Vermont Proctor Maple Research Center

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Mary An Godshall Sugar Processing Research Institute, Inc.

#### Steven Lloyd

United States Department of Agriculture Agricultural Research Service, Southern Regional Research Center

oncentrating maple sap with reverse osmosis (RO) significantly ✓increases the efficiency and profitability of processing sap into syrup by reducing the amount of both fuel and time required to concentrate the material to syrup density in the evaporator. However, because this also reduces the amount of time sap is processed in the evaporator, and since most of the reactions from which the flavor and color properties of maple syrup are ultimately derived occur as sap is processed with heat in the evaporator, it is possible that this could result in impacts on the finished syrup, causing it to differ from syrup produced with raw sap. with commercial maple equipment to investigate the potential effects of the use of RO on the composition, properties, and flavor of the maple syrup produced. The following is a brief, general summary of these experiments and the results observed. More comprehensive descriptions of each study can be found in the scientific journal articles published for each.

All experiments were conducted in the Maple Processing Research Facility at the University (MPRF) of Vermont Proctor Maple Research Center in Underhill Center, Vermont (UVM-PMRC) (Figure 1).

#### Maple Digest 54(3): 11-33

# Higher concentration levels: >30%

#### Newest RO Technology: Pre-concentrates sap to 30-40%



CDL 30+ RO H<sub>2</sub>O Innovation Super-Concentrator<sup>™</sup> Lapierre HYPERBRIX<sup>™</sup> Memprotec



### **Study Objective**

What are the impacts of ultra-high RO concentration on syrup **flavor**?



#### Sensory Evaluation Experiment:

Is the flavor of syrup produced with sap concentrated to 30-40 °Brix appealing and liked?

Is the flavor characteristic of pure maple syrup?

#### **Syrup Samples**

2016 Season 6 producers using High-Brix 3 samples each: Early, Middle, Late

%LT, °Brix, Organic vs Conventional

Matched Control Samples Produced with standard RO (no steam, air injection, etc.)

Matched for Color/Grade, Organic/Conventional





# **Sensory Evaluation Experiment**

46 panelists recruited and pre-screened:

Like maple syrup Non-smokers Familiar with current grading system

4 Experiment sessions



# **Sensory Evaluation Experiment**

# How much do you like or dislike the **overall flavor** of this syrup?

9-point hedonic scale Dislike extremely  $\rightarrow$  Like extremely

1. Please indicate how much you like or dislike the overall flavor of this syrup:         Image: State of the synup is the synup of the synup is the synup of the synup o	Sample I	D#							
Dislike Dislike Dislike Neither Like Like Like very Like	1. Please	indicate h	ow much yo	u like or d	islike the o	overall fla	<b>vor</b> of this s	yrup:	
Dislike Dislike Dislike Dislike Neither Like Like Like very Like extremely very much moderately slightly like or slightly moderately much extremely									
dislike	Dislike extremely	Dislike very much	Dislike moderately	Dislike slightly	Neither like or dislike	Like slightly	Like moderately	Like very nuch	Like extremely

(Is flavor of syrup made with High-Brix liked?)

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# **Sensory Evaluation Experiment**

#### How much do you agree or disagree with this statement: "The flavor of this syrup is characteristic of pure maple syrup"

7-point Likert scale Entirely disagree  $\rightarrow$  Entirely agree

2. Please indicate how much you agree or disagree with this statement: "The flavor of this syrup is characteristic of pure maple syrup."

Entirely disagree	Mostly disagree	Somewhat disagree	Neither agree or disagree	Somewhat agree	Mostly agree	Entirely agree

(Is flavor of syrup made with High-Brix characteristic of pure maple syrup?)

#### Results

#### Is the flavor of syrup produced with High-Brix processing liked?

Indicate how much you like or dislike the overall flavor of this syrup



Overall average percentage of responses in each category across Control (n=6) and High Brix (n=9) samples.



#### Results

Is the flavor of syrup produced with High-Brix characteristic of pure maple syrup?

Indicate how much you agree or disagree with this statement: "The flavor of this syrup is characteristic of pure maple syrup."



Overall average percentage of responses in each category across Control (n=6) and High Brix (n=9) samples.



# **Preliminary Conclusions – High-Brix**

Syrup made with High-Brix processing has flavor that is generally liked and characteristic of pure maple syrup

"Liking" and "characteristic of maple syrup" similar to syrup of the same color/grade made with standard RO processing

Flavor consistent with syrup of similar color grade

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# Thank you!

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\*\*Producers\*\*
\*\*Sensory panelists\*\*

