Development of a Benchtop Method to Polymerize Lactose to Soluble Fiber

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Introduction

Extrusion has been utilized for the polymerization of sugars into larger oligosaccharides.[1,2] The high temperature, pressure and shear from the extruder allows for continuous chemical synthesis of oligosaccharides.[2] A mixture of lactose, glucose and citric acid has been polymerized using a twin-screw extruder to yield polylactose.[3] However, previous research in our lab demonstrated that the lactose in permeate and acid whey could not be polymerized using our standard extrusion method. Inhibition factors for this polymerization reaction are not currently well understood. The impact of the citric acid content and extruder feed rate have been researched[3], yet chemical properties such as moisture and mineral content have not.

Objective

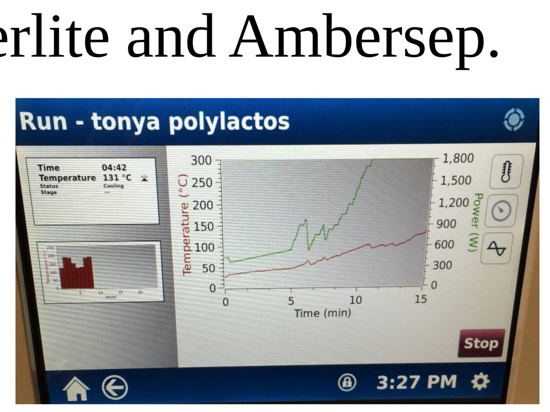
The objective of this study was to develop a benchtop method for polymerization to identify reaction inhibition factors before extrusion scale up.

Materials and Methods

<u>Sample Preparation:</u> 7 grams of a blend containing citric acid (6%), lactose (74%) and glucose (20%) were added to 8 Teflon MarsXpress vessels. The vessels were closed and the product equilibrated for 48 hours when water was added.

<u>Heating in the Microwave Reactor:</u> The vessels were heated simultaneously in a CEM Mars 6 microwave reaction system. All vessels were continuously monitored for temperature via an internal infrared thermometer.

<u>Polymerization Confirmation:</u> HPLC-ELSD was used for polymerization confirmation. Cooled samples were prepared for analysis by dissolving product in water to a concentration of 50 mg/ml and then passing the solution through ion-exchange resins made of Amberlite and Ambersep.



Heating Profile Development

1. Original Heating Profile:
- 5 minute ramp time to a 180°C set temperature
- 3 minute 30 second hold time

Observations: The product melted despite the uneven heating between vessels. Even though the product set temperature was not reached, polymerization was observed.

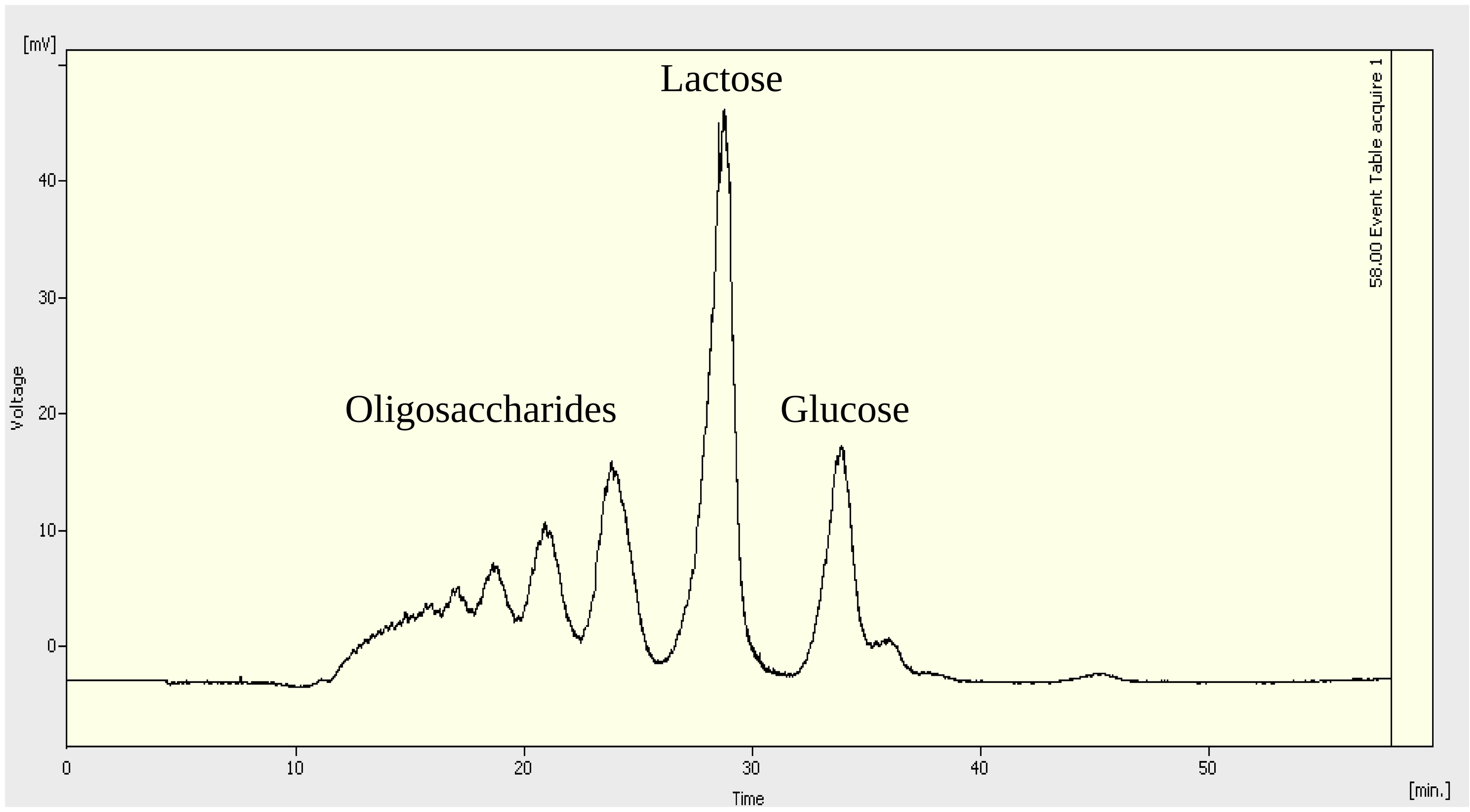
2. Modified Heating Profile:
- Extended ramp time to 15 minutes
- Decreased set temperature to 150°C
- Decreased hold time to 1 second

Observations: Uneven heating occurred between the vessels and the product reached a temperature that was too high (~200°C). The resulting product was black and burned.

3. Final Modified Heating Profile:
- Maintained ramp time of 15 minutes and hold time of 1 second
- Decreased set temperature to 140°C
- Added <1% (w/w) water to each vessel before heating

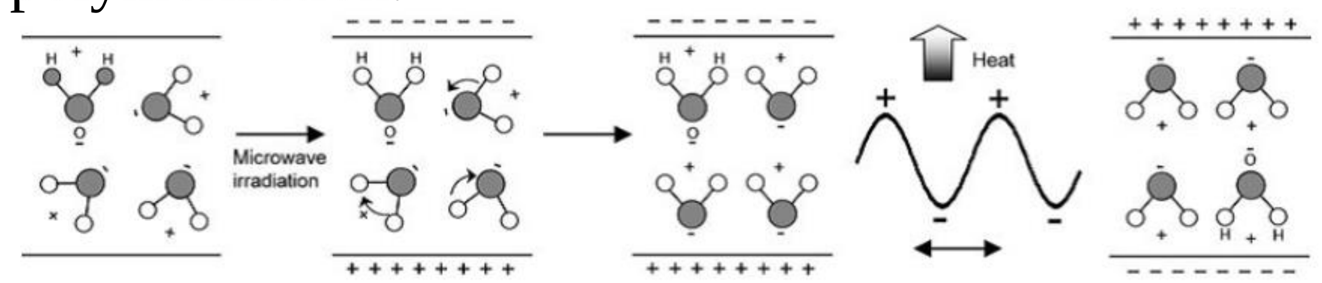
Observations: The product melted and even heating was observed between vessels. The resulting product was successfully polymerized

Polymerization Confirmation from Final Heating Profile



Discussion

- Melting and polymerization of the lactose was possible at a lower temperature than the extrusion method.
  - The Mars 6 system was under higher pressure than the open extruder system.
- An increase in ramp time prevented large power surges at the end of the heating cycle that helped reach the product set temperature.
- The addition of a small amount of water was necessary to consistently achieve the target temperature in all vessels for every run.
  - Addition of a polar solvent increased dipole rotation due to the microwave energy, creating more even heating.
  - Excess water could cause hydrolysis instead of polymerization.



Saxena and Chandra, 2011

Future Work

- Optimize water addition for the final heating profile.
- Investigate the impact of moisture and mineral content on lactose polymerization to mimic acid whey composition.
- Scale up the learnings via extrusion in the pilot plant.

Acknowledgements

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References

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