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INTRODUCTION

■ Agriculturally sourced (corn/sorghum) bioethanol production produces a lipid slurry called bioethanol production side stream. This lipid slurry is often **wasted** or used as animal feed.



ISSUES

- Approximately 0.7 million lbs. of corn/sorghum side stream lipid is produced yearly, which is rich in free fatty acids, triacylglycerols, and high-melting point waxes.
- The presence of high amounts of lipids with similar solubilities makes the fractionation challenging.
- Due to the complex matrix of the bioethanol side stream lipids, it is not effectively utilized by any industry.

PROPOSED SOLUTION

- Using a novel and green approach based on supercritical carbon dioxide (SC-CO₂) to fractionate triacylglycerols and free fatty acids from high melting point waxes

GOAL AND OBJECTIVES

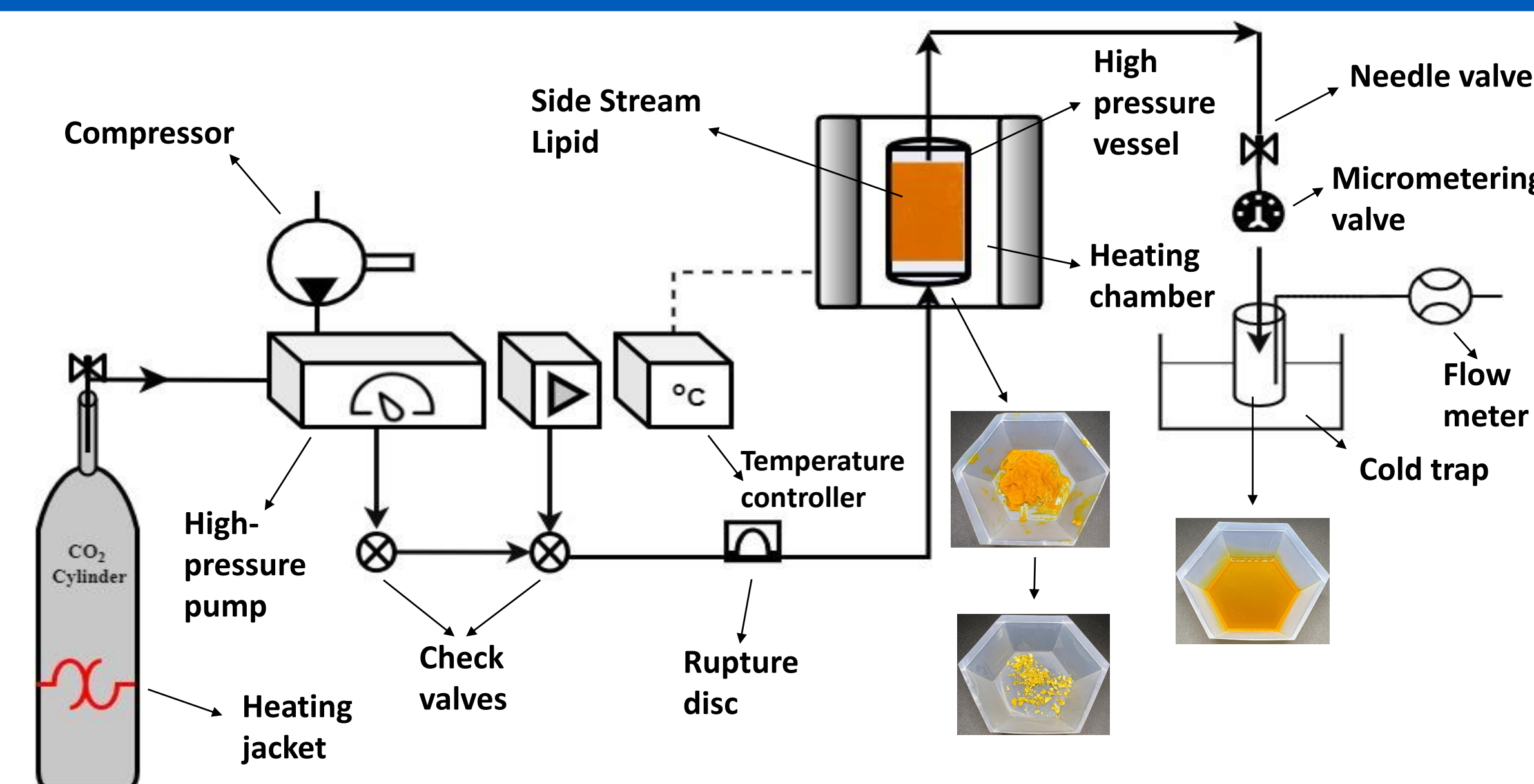
Goal:

- To develop a novel, green process based on SC-CO₂ to selectively extract and fractionate lipids present in the stillage (corn/sorghum mixture feed to the fermenter) to purify high-melting point waxes.

Specific objectives:

- To develop a statistical model optimizing SC-CO₂ conditions for maximum free fatty acids/triglycerides extraction leaving behind high melting point waxes.
- Generate a second order equation explaining the variation in the process of lipid/wax fractionation by SC-CO₂ to predict future operations.

PROCESS SCHEMATIC DIAGRAM

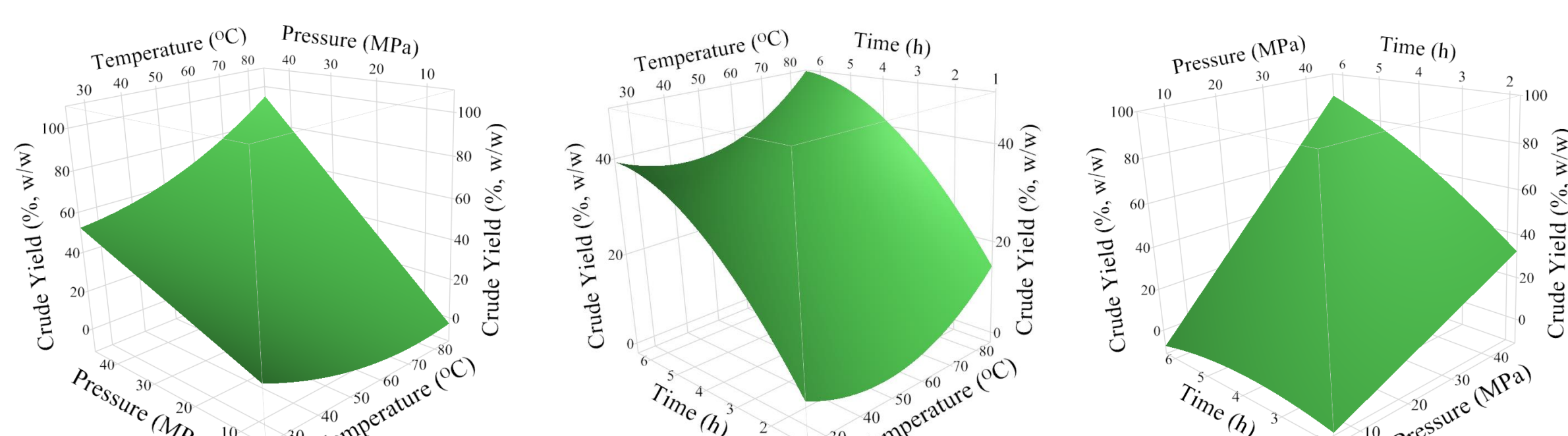


DESIGN OF EXPERIMENT

- A 3-factor (3-level each) Response Surface Box-Behnken Design was created to optimize the procedure for a **MAXIMUM OIL** and **MINIMUM WAX**.

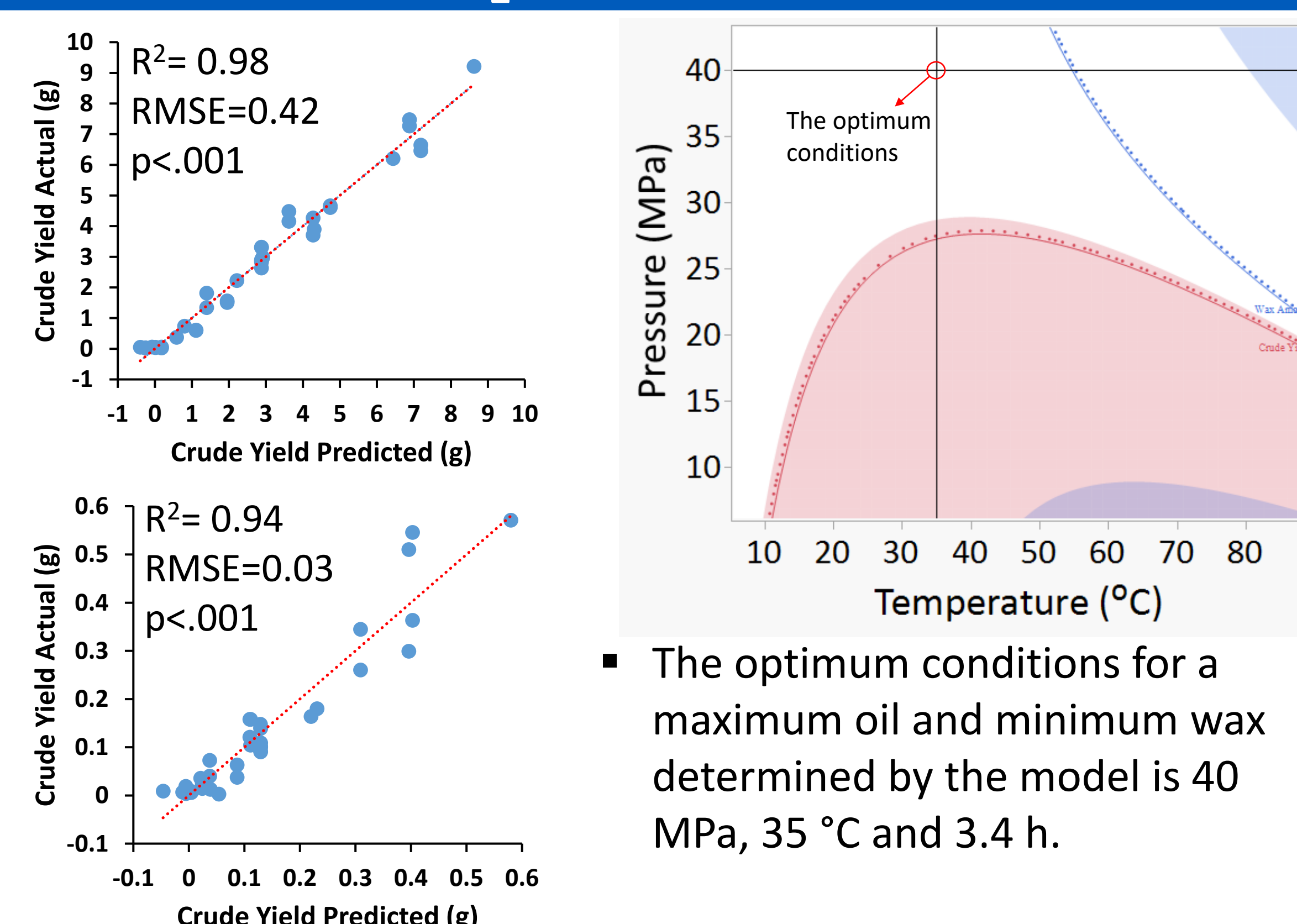
Variable	Level		
	-1	0	1
Temperature (°C)	35	55	75
Pressure (MPa)	8	24	40
Time (h)	2	4	6

RESULTS



- The statistical analyses show that all three variables (pressure, temperature, time) and their interactions are significant ($p < 0.05$).
- The model suggests that operating at high pressures (30 – 40 MPa) and low-moderate temperatures (35 – 50 °C) for at least 3 h maximizes the oil extraction while limiting the wax recovery.

OPTIMIZED SC-CO₂ FRACTIONATION CONDITIONS

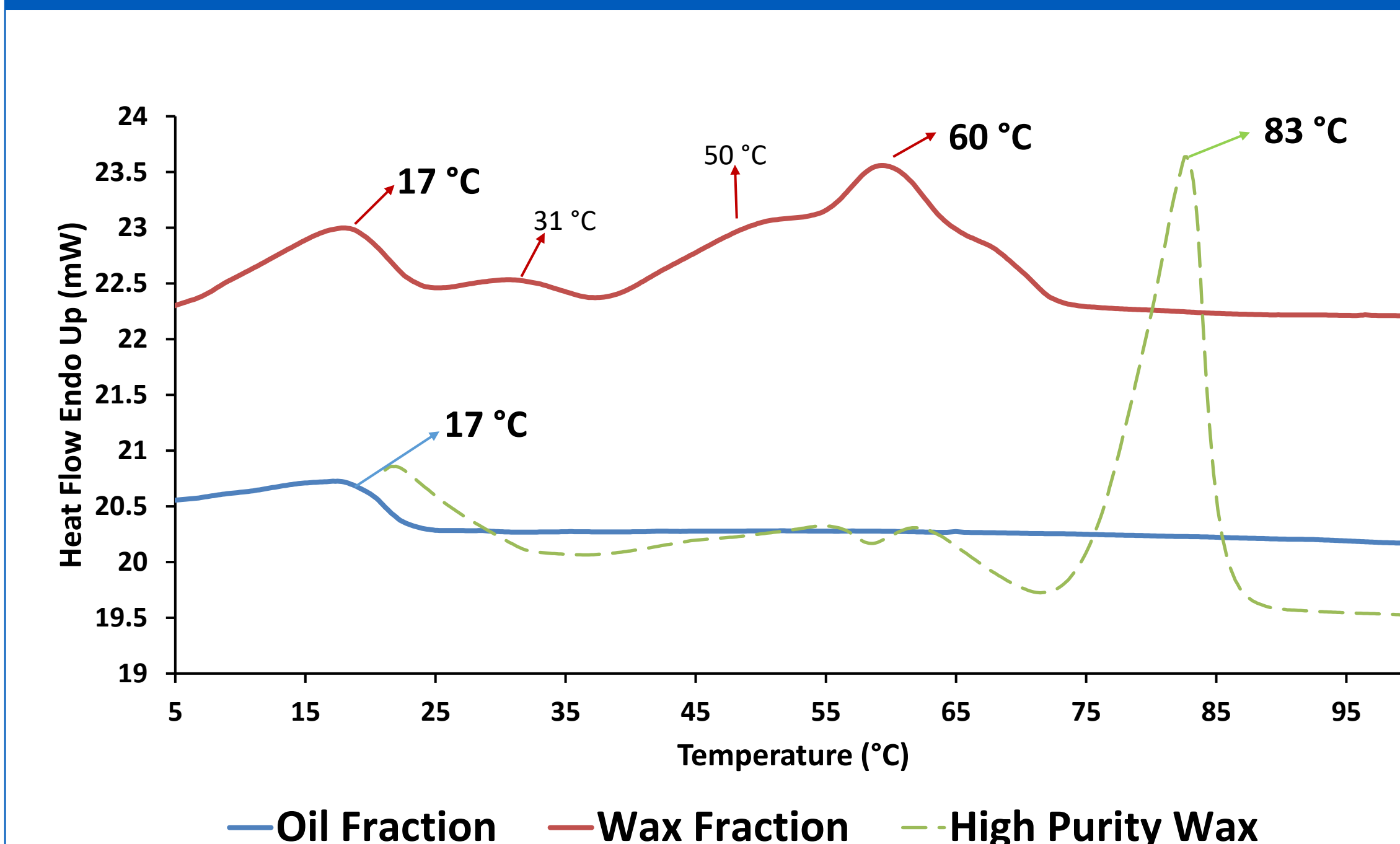


- The optimum conditions for a maximum oil and minimum wax determined by the model is 40 MPa, 35 °C and 3.4 h.

MODEL VERIFICATION

- Oil fraction that is liquid at 23 °C.
- Wax fraction that is solid/creamy at 23 °C.
- High purity wax fraction that is solid at 23 °C.
- When the side stream was processed at the optimized conditions (40 MPa, 35 °C and 3.4 h), 4.1 g oil was recovered from 10 g crude corn/sorghum side stream lipid with only 95 mg wax content, **as predicted by the model**.
- The model also predicted that SC-CO₂ at 40 MPa, 75 °C and 6 h generates high melting point (83 °C) wax pellets.

MELTING POINTS OF THE WAXES



CONCLUSIONS

- Following the equations generated by the statistical model, oils (melting point of 17 °C) were selectively extracted from the crude side stream lipid.
- As the oil is extracted from the crude lipid, the wax fraction that remains in the vessel has mild melting point.
- The model was also shown effective in recovering high purity wax pellets from the crude side stream lipids.

INDUSTRIAL APPLICATIONS

- A green and sustainable way of utilizing an agro-industrial waste as a source of high purity oil for the biodiesel industry and high-value wax for the food industry.
- A food-grade scalable method to extract waxes with desirable qualities for food coatings and cosmetic products.
- Alternative source for beeswax (melting point 60-62 °C) and carnauba wax (melting point 84 °C).

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