Investigation of Process Variables in Supercritical Carbon Dioxide Extraction of Soybeans

Using a Teledyne ISCO Syringe Pump and Extraction Module

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Syringe Pump Application Note
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Abstract and Introduction

Soybean oil is one of the most widely used plant oils today. Most soybean oil is extracted using hexane solvent extraction; while being fast and efficient, this has several disadvantages. Supercritical CO_2 offers an attractive alternative for extraction with smaller environmental and health impacts because it is nontoxic, nonflammable, cheap, and readily removed from the extract. Our research is focused on optimizing processing conditions to reduce the energy cost to extract soybeans, including the amount of supercritical CO_2. The Carbon Recycling Center in the Department of Biological Engineering at the University of Missouri has a Teledyne ISCO 260D [see note] syringe pump and an SFX-2-10 extraction module. This system was used to perform all of the experiments, providing precise control of pressure and flow of supercritical CO_2 through the soybean meal.

Experimental Procedure

These experiments were conducted with Teledyne ISCO model 260D syringe pumps in combination with an Teledyne ISCO SFX 2-10 and SFX 220. Only one supercritical fluid extractor was used at a time. Each pump can achieve a maximum pressure of 51.7 MPa and the maximum flow rate is 100 mL/min; however, two pumps were used in parallel to achieve higher flow rates. The two pumps in parallel could supply CO_2 at a rate > 150 mL/min. Each pump was connected to a high-pressure CO_2 reservoir tank with 99.9% purity.

Through carefully monitoring conditions in the syringe pump (i.e. temperature, pressure), the amount of CO_2 pumped to the extractor can be determined. The $scCO_2$ to be pumped was held at a constant temperature of 21.5 °C by a water jacket on the syringe pump. The Peng-Robinson equation of state was used to calculate CO_2 properties. At 21.5 °C and 48.3 MPa, $scCO_2$ has a density of 1.096 g/mL, which was used to determine mass flow rates. The unit was run in constant pressure mode, which kept the pressure at a set value by changing the flow rate within a small range.

The output of the syringe pump was fed into either an SFX 2-10 or SFX 220 supercritical fluid extractor. It consisted of a heater with temperature controller and dual extraction chambers. Capillary tubing (standalone fused silica tubing with 0.1 mm inner diameter) and a valve controlled the output flow rate exiting the extractor. The SFX 220 allowed for automated control through the pump controller and granted finer control over the mass of CO_2 extracting the soybeans. Therefore, the majority of the experiments were conducted with the SFX 220 extractor.

A typical experiment involved loading about 4 gallons of flaked soybeans into a 10 mL extraction chamber consisting of a metal cylinder with two caps, each of which holds a filter between it and the cylinder. The supercritical carbon dioxide was allowed to equilibrate with the temperature of the water jacket. The extraction vessel was placed in the extraction chamber and allowed to equilibrate to the extraction temperature and pressure. Then, the supply valve was opened to allow the filling of the extraction chamber. The extract valve was then opened to allow the flow of the desired amount of $scCO_2$ through the extraction vessel. Extraction percent and mass balances were determined gravimetrically.

This setup created a semi-continuous system, which allowed for the assessment of large-scale continuous systems. Because of the high accuracy of the pump output flow rates, average solubility can be accurately determined. Dividing the amount of oil extracted by the mass of CO_2 used yields the average solubility inside the extractor. Regressing back to a zero mass of CO_2 yields a pure solubility at the extraction conditions inside the system. This information gives a best case scenario for your system and allows for efficiency calculations.

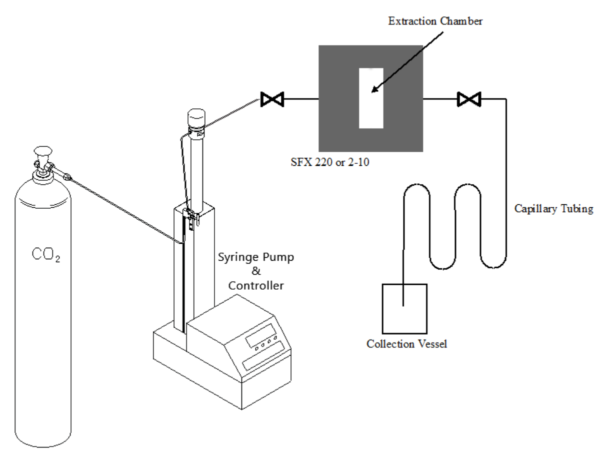

Figure 1: Experimental setup for extracting soybeans with supercritical CO_2

Precise amounts of CO_2 at constant pressure can be delivered with a 260D pump.

Results

A reference in the literature from our lab has more figures describing the effect of different processing conditions on extraction (Wilkinson 2012). Important extraction parameters were explored at 80°C and 48 MPa without the use of co-solvents. This study found the presence of hulls had no effect on extraction efficiency, showing the de-hulling step can be removed for production of 44% protein soybean meal after extraction. Extraction at 80°C and 48 MPa resulted in a lower solvent to meal ratio (< 10 g solvent/g meal) required for extraction than previous results at lower temperatures. Residence times of 60 s are sufficient for complete extraction. The residence time allows for determination of maximum flow rates through different size extraction chambers. This data can be used in designing an efficient, high-throughput extraction apparatus that can replace conventional extractions with a simpler, less intensive process. Our results and the literature point to extractions at much higher pressures, while at elevated temperatures for better performance.

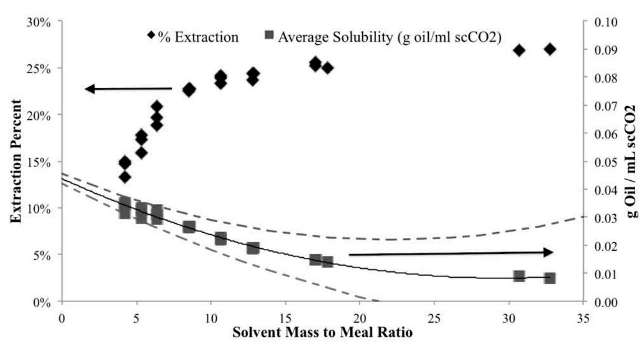

Figure 2: Effect of solvent to meal ratio

Percent extraction and oil solubility with 95% confidence intervals as a function of mass supercritical CO_2 flowing at the same flow rate through the soybean meal.

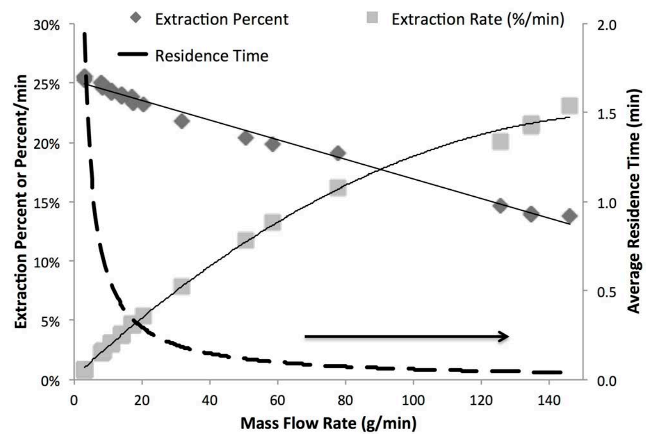

Figure 3: Effect of mass flow rate

The effect of the flow rate on extraction percent and the extraction rate of oil from the soybean at extraction parameters of 87 grams $scCO_2$, 48 MPa, and 80°C.

Conclusion

Extraction of triglycerides was explored at 48 MPa and 80 °C. Specific parameters that were studied include solvent flow rate, solvent mass to meal ratio, solvent residence time, particle diameter, and presence of soybean hulls. A Teledyne ISCO syringe pump and extractor were used to precisely control the pressure and flow of supercritical CO_2 through the soybean meal. The use of the higher temperature in this study, extraction efficiency was improved, requiring smaller residence times and solvent mass to meal ratios. This process has applications in both the food and biofuel industries for generation of soybean oil.

References

1. N. Wilkinson, R. Hilton, D. Hendry, C.Venkitasamy, and W. Jacoby. “Investigation of Process Variables in Supercritical Carbon Dioxide Extraction of Soybeans.” In Press at *Food Science and Technology International*, 2013.

Note:

The 260D model pump, which was used during the original experiment, is discontinued. Current model 260x is the recommended replacement for the older 260D model.

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Product model names have been updated in this document to reflect current pump offerings.

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