FINAL REPORT
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CARBON DIOXIDE ENHANCED PRESSING FROM RENDERED MATERIALS

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Lay Summary: The goal of this work is to investigate the use of carbon dioxide (CO_2) as a green solvent for applications in the rendering industry. The potential impacts of this work revolve around creating value-added co-products for energy, consumer products, and commodity chemicals/materials. This research has focused on the use of supercritical and liquid CO_2 to enhance the fat separation during the mechanical pressing of rendered materials at industrial relevant scales. Our prior work demonstrated the ability to enhance the mechanical expression of fat from a ground poultry meal where up to 81% of the residual fat in the poultry meal was recovered by mechanically pressing the poultry meal in the presence of supercritical CO_2.[1-2] In other words, the fat content of the ground poultry meal was reduced from the initial 12.1% to a final 2.3% after pressing in a lab-scale batch press operating at 40°C, 3000 psi of CO_2, and 8700 psi mechanical pressure. Based on these results, we have conducted pilot scale studies at the Crown Iron Works R&D Test facility in Roseville, MN using the HIPLEX System. Results from this work have demonstrated the feasibility of applying the HIPLEX technology to enhanced pressing of poultry crax at industrial scale.

Objective (s):

The goal of this work is to explore the extent to which the HIPLEX system can be used to enhance the fat recovery from meat and bone meal (MBM). Specific objectives of this work will include:

1. Determining the optimal operating parameters for HIPLEX with MBM crax and ground MBM containing 14% fat.
2. Correlating the operating parameters for HIPLEX with fat expression yield.
3. Determining the impact of CO_2 pretreatment of the MBM crax before pressing.

Project Overview:

The goal of this work was to investigate the use of carbon dioxide (CO_2) as a green solvent for applications in the rendering industry. The basic process of rendering consists of 1) size reduction, 2) cooking with high temperature steam, 3) pressing to separate fat, and 4) grinding of protein.[1] Within this scenario, there are opportunities for enhanced separations that will preserve value added content within the rendered material, as well as provide more efficient separations. This research focused on the use of supercritical and liquid CO_2 to enhance the fat separation during the mechanical pressing of rendered materials. Our prior work has demonstrated the ability to enhance the mechanical expression of fat from a ground poultry meal where up to 81% of the residual fat in the poultry meal was recovered by mechanically pressing the poultry meal in the presence of supercritical CO_2.[2] In other words, the fat content of the ground poultry meal was reduced from the initial 12.1% to a final 2.3% after pressing in a lab-scale batch press operating at 40°C, 3000 psi of CO_2, and 8700 psi mechanical pressure. More recently, our work has been focused on exploring the scalability of these methods to continuous pressing at an industrial relevant scale. We performed pilot scale tests of the CO_2 enhanced pressing of rendered materials at the Crown Iron Works pilot scale test facility in Roseville MN using their HIPLEX system.

CO_2 is an attractive extraction solvent because it is non-toxic, chemically inert, inexpensive, abundant, and FDA approved. From a processing and separations standpoint, CO_2 is attractive because the solvent properties can be tailored by controlling the temperature and pressure. We have previously demonstrated the ability to effectively extract fat components from rendered material using CO_2 on a laboratory scale. The tunable properties of the CO_2 are most beneficial as an extraction media in the recovery of the fat extract, which can be achieved by simply altering the temperature and pressure, resulting in a facile and economic separation and CO_2 recycle. This is in contrast to traditional methods of fat extraction that requires high temperature extraction with a volatile organic solvent, such as hexane. These solvents are volatile, flammable, often not approved by the FDA, and require distillation to recover the solvent from the fat extract. Furthermore these solvents are not ideal for specific component extraction.

Another unique property of CO_2 is its antimicrobial properties, which has been implemented in a variety of sterilization applications in the food and medical industries.[3] Work in this area has demonstrated that simultaneous exposure to CO_2 with heat is more effective in sterilization than either technique independently. Supercritical CO_2 has the ability to kill most microorganisms and to inactivate viruses, including human pathogenic strains, which has led to sterilization and pasteurization applications where conventional methods prove ineffective or not feasible. Specific examples include the inactivation of E. coli[4-6], Salmonella[4, 7], bacterial spores[8], S. aureus and other microorganisms with applications ranging from medical devices and textiles[9] to palm oil[10], pork meat[4], and sewage sludge[5].

Motivation for this work is driven primarily by markets for new rendered products. Historically the market for fats and proteins have been similar, however there have been instances where the value of fats have increased to \$0.40 - \$0.50 per pound versus \$0.16 - \$0.20 per pound for proteins. Furthermore, as our economies move further away from a dependence on petroleum products, the precedence is growing for chemicals and additives derived from alternative renewable resources. Emerging markets are developing for a lower fat content meals for pet foods where the extracted fats could be applied to the surface of the food pellets and increase the palatability while maintaining a low fat pet food. Current press operations produce rendered materials that contain around 12% fat content. It is our contention that the amount of fat extracted from rendered materials can be increased while potentially increasing the value of the meals.

This research is a continuation of our prior research on the use of CO_2 to enhance the recovery of fat components from rendered materials. We demonstrated the ability to reduce the fat content in poultry meal and meat and bone meal down to less than 1% in a semi-batch extraction process.[2,3] We have also applied thermodynamic models to the extraction process, which could be used for process simulation and economic analysis. We constructed a press apparatus that enables the application of mechanical force while under CO_2 pressure in order to examine the enhanced fat expression from the rendered materials. Results from this work demonstrated that CO_2 assisted mechanical expression of fats from rendered material could be used to obtain a yield of 81%. This yield represents a reduction in the remaining fat content from 12.1% to 2.3%. The optimal conditions for a maximum extraction in the range of conditions were 40°C, 3,000 psi of CO2, and 9,000 psi of effective mechanical pressure.[3]

Building upon these results we have extended our studies to the pilot scale where CO_2 enhanced pressing will be performed in a continuous press at a rate of 1 ton per hour. These pilot scale experiments were performed at the Crown Iron Works pilot plant facility in Roseville MN. The primary collaborator at Crown is Chas Teeter. At the pilot plant, Crown has a demonstration unit referred to as High Pressure Liquid Extraction (HIPLEX). HIPLEX is a mechanical screw press designed to inject liquid CO_2 into the press to enhance the oil recovery from seeds. Crown developed this technology with Harburg-Freudenberger (HF) and it is currently proprietary to HF presses, however recent negotiations have resulted in the split of Crown and HF. This was in part a result of Crown Iron obtaining Skett Presses. Theoretically, the HIPLEX process can be applied to other press manufacturers. To date, the HIPLEX technology has been geared to seed oil recovery and the application to rendered materials is transformative.

Materials and Methods: Planning for this project began in April 2014 for pilot-scale trials to test the CO_2 enhanced pressing of rendered materials at the Crown Iron Works test facility in Roseville MN using their HIPLEX system. HIPLEX is a mechanical screw press designed to inject liquid or supercritical CO_2 into the press to enhance the oil recovery from seeds, see Figure 1. Crown developed this technology with Harburg-Freudenberger (HF). The first round of pilot scale experiments were conducted during the week of July 27 to August 1st, 2014 with 3 days of testing on two types of poultry material: a) A high-fat green poultry crax and 2) a pressed poultry crax. These materials were obtained from a local Sanimax rendering plant in South St. Paul MN that is located less than 20 miles from the Crown pilot plant. Tim Guzek, Brian Argo, and Tim Kedrowski (Plant Manager) of Sanimax were critical in our ability to obtain the rendered materials for this study.

The HF PIPLEX press is shown in Figure 1 where the rendered material is fed into a variable speed screw feeder into a jacketed screw conveyer that was used to heat the material with indirect steam. The material was then fed through a series of conveyors to the inlet of the H-F press (above the ladder shown in front of the blue H-F press). The material then traveled through the press, exiting on the right side and conveyed to collection totes on the far right.

We tested the Sanimax green poultry crax (42% fat by hexane extraction) on July 29th and the Sanimax pressed poultry crax (16.2% fat by hexane extraction) on July 30th. The target RM feed rate was 8 lb/min and the CO_2 feed rate was varied. The experimental design and experimental run parameters are detailed in the prior reports.

Overall, this first set of experiments had limited success. Admittedly, this is the first attempt to work with rendered materials for the staff at Crown Iron Works, and despite a lot of planning, prior examination of the feed materials, and confidence of the staff, the trials ended up being a learning experience. Results from the initial trials prompted significant redesign in the press configuration including:

- Rebuilding of the press cage and adjustment of the lining bars.

- Installation of a horizontal cooker and modified conveyer into the press to better feed the material at temperature into the press (>260°F). Fig 2A and 2B
- Installation of additional temperature probes to better monitor the behavior inside of the press. 50% more probes. Fig. 2C
- Five different modifications to the press shaft to optimize fat removal for the rendered materials. Fig 2C

The five successive shaft modifications were done in collaboration with the HF press manufacturers in Germany, who are also interested in the research. The goal was to better feed the rendered material through the press, which requires a more gradual taper in the shaft. Modification #2 and modification #3 both contained a gradual taper but they were unable to create the RM plugs that are required to maintain CO_2 pressure in the press. Modification #4 was close to spec and a final modification #5 performed best. These shaft modifications were most closely modelled after a shaft configuration for poultry crax with the added ability of creating the plug seals required to maintain CO_2 pressure. Shaft modification #5 was tested on March 5th and 6th, 2015 on high oil content seed, which closely resembles the poultry meal.

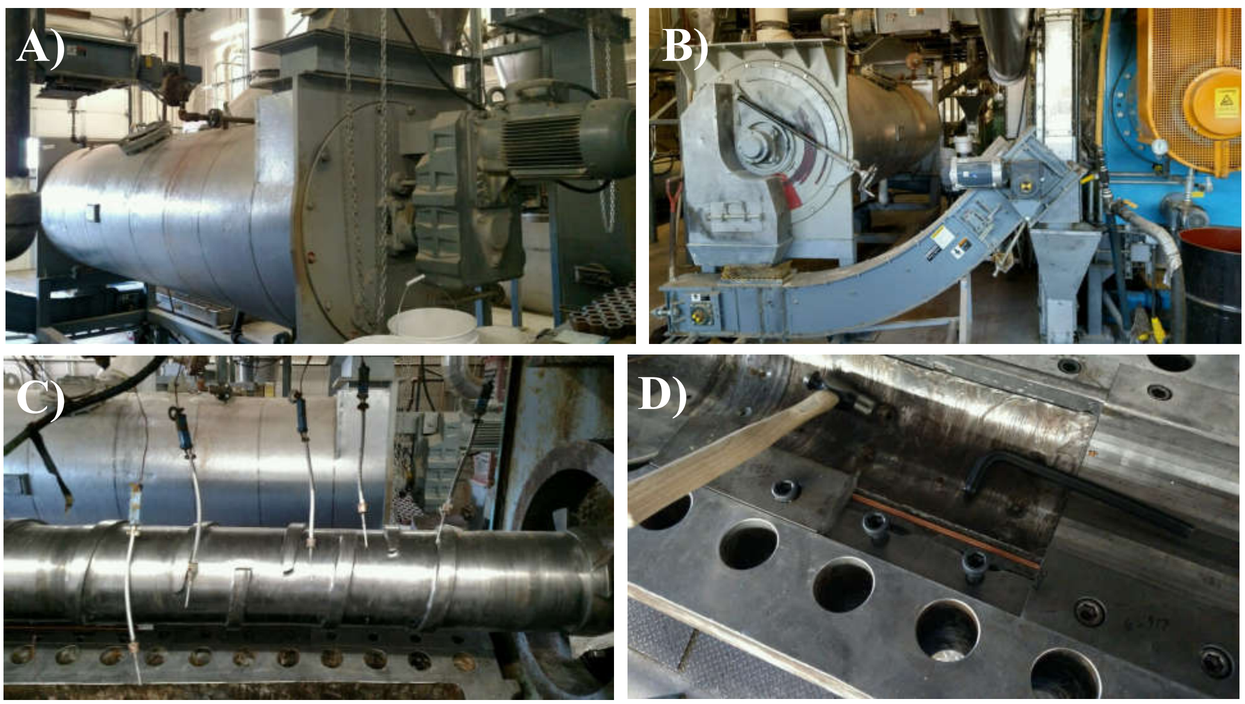

Figure 2. Modifications to the HIPLEX system A) inline cooker to heat rendered material to temperature, B) conveyer system to feed material into HIPLEX, C,D) redesigned shaft and cage configuration with additional temperature probes.

On April 15th, 2015, trial tests were conducted by the staff at Crown on green poultry crax and pressed poultry crax with the modified press configurations with favorable results which led to three additional days of trials on June 17th – 19th, 2015. These trials included testing Green Poultry Crax (left) and Green MBM Crax (right), shown in Figure 3. Results from these trials are detailed below.

Results and Discussions: The reconfigured HIPEX press was successful in pressing rendered materials in the presence of CO_2 demonstrating feasibility. Results from the April 15th, 2015 trial demonstrated a significant reduction in the fat content for the green poultry crax from 17%

fat without CO2 and 7.5% fat with CO_2. The pressed crax were pressed to 7% without CO_2 and then down to 5.5% with CO_2. These results were single point tests conducted by the staff at Crown that demonstrated proof of concept as well as the potential enhancement in pressing efficiency with the use of CO_2. The results for the green and pressed crax were both positive, showing enhanced pressing, however the green crax results were most significant and motivated further study.

On June 17th, the green poultry crax were run successfully but issues with the CO_2 delivery system prohibited further study. On June 18th, the CO_2 system had been repaired and successful trials were conducted with the green poultry crax. The poultry crax were fed at a rate of 9.5 to 11 lbs/min into the cooker. The steam feed to the cooker was regulated to bring the poultry crax to a temperature of 250-260°F. Samples were collected for the pressed crax without CO_2. CO_2 was then introduced at a rate of 4 lb/min or a 1:1 ratio of fat to CO_2 on a mass basis. This CO_2 feed rate was too high and resulted in the material plug seals "blowing out". The CO_2 flow rate was reduced to 1.5 lb CO_2 / min or roughly a 3:1 ratio of fat to CO_2 and samples were collected running with low temperature CO_2 (30°F) and high temperature CO_2(185°F). At this point, the press stopped feeding material because it was too "wet" due to excess fat being fed into and accumulating in the cooker. The free fat in the bottom of the totes was an issue and was drained from the totes prior to feeding into the cooker. The crax feed rate was increased to 30lbs/min and high temperature CO_2 at 4 lbs/min. This resulted in feed issues compounded by free fat pooling in the cooker and saturating the feed to the press. To alleviate this, the cooker was bypassed and the crax were fed without issue at 20lbs/min. Samples were collected without CO_2, with low temp. CO_2, and with high temp. CO_2. Results from these runs are detailed in Table 1.

On June 19th 2015 we ran MBM green crax. The large bone pieces caused issues with the feed auger as well as with the conveyer from the cooker to the press. It was necessary to feed the material by shovel at a steady rate onto the conveyer and bypass the cooker. The MBM green crax were fed at a rate of 20 to 25 lbs/min. Samples were collected without CO_2, with low temp. CO_2 (2 lbs/min), with high temp. CO_2 (2 lbs/min), with low temp. CO_2 (2 lbs/min), and without CO_2. Results from the MBM green crax are detailed in Table 1.

Figure 3. Rendered materials used in the pilot trials. A) Poultry green crax and B) MBM green

crax.

Table 1. Results from pilot trials at Crown Iron Works on June 18-19, 2015.**Error! Not a valid link.** Two set of results were obtained for the green poultry crax which show the effects of CO_2 pressure, temperature and amp load on the resulting fat content. The most significant result is that the fat content was 2.7% lower (13.8% to 11.1%) with the use of heated CO_2 at similar operating parameters, a 20% fat reduction. This 20% reduction in fat content was achieved with a load of 75 amps on the press and a constant Cake temperature of 250 °F. The heated CO_2 consistently performs better than the CO_2 that is not heated. When the high pressure CO_2 undergoes rapid expansion in the press, the Joule-Thompson effect results in significant cooling. To counteract this, the CO_2 is heated to a supercritical state at 185°F prior to injection. Use of liquid CO_2 at 30°F can on instance result in lower CO_2 pressing efficiency (a 5% reduction from 13.8% to 13.1%) due to the lower temperature effect.

Analysis of the green poultry crax results also indicate that the addition of CO_2 can also decrease the amperage load on the press, which translates to less energy consumption by the press and less heat generation due to the friction between the rendered material and the press shaft. The lower cake temperature is important because local hot spots at the shaft interface can lead to charring of the rendered material, which degreases the nutritional content. The second set of poultry crax numbers show that with the use of heated CO_2, lower fat content (7.4% vs 8.1%) can be achieved with significantly lower force applied (65 amps vs. 95 amps). The ability to press the fat from the crax is a balancing act in terms of the operating parameters. Increased amperage can result in lower fat content but requires more energy and can result in cake charring. Temperature effects are also significant where higher temperature can result in lower fat content but again, cake charring can be an issue. Our results demonstrate that the use of heated CO_2 mitigates the cooling effects and enables increased pressing efficiency at decreased loads.

The results from the MBM pilot trials were not favorable and showed higher fat content with the use of CO_2. This can be attributed to several factors. First, the press configuration is set up for poultry and not MBM. Similar results were also obtained with our first poultry crax trials but following the press reconfiguration, favorable results were obtained. Second, the material feed system was not set up to accommodate the large pieces of bone in the MBM which led to inconsistencies. We believe that similar favorable results can be achieved with MBM crax by designing a press configuration and material conveying system that is appropriate for the MBM. It is known in the industry that there are specific differences in the press configuration and shaft design that are more favorable for pressing MBM versus poultry crax. Based on these factors, we are not placing a large amount of confidence in these results for MBM.

Conclusions: There is significant potential and need for improved methods of isolating fats from rendered materials. This work is unique in its ability to be incorporated into existing operations without significant disruption and provides potential innovation in a process that has not seen any in several decades. The ability to press the fat from rendered materials is a balancing act in terms of the operating parameters. Increased amperage can result in lower fat content but requires more energy and can result in cake charring. Temperature effects are also significant where higher temperature can result in lower fat content but again, cake charring can be an issue. Our results demonstrate that the use of heated CO_2 mitigates the cooling effects and enables increased pressing efficiency at decreased loads. This is a significant advancement and holds promise for potential application. The ability to increase fat yields by 20% with the use of CO_2 in a continuous, industrial scale process is significant. Furthermore, the ability to obtain a low-fat meal without the use of solvent extraction is also significant, particularly if a fat content of 6% is achievable without degrading the nutritional content.

References:
[1] Meeker, D.L., ed. *Essential Rendering: All about the Animal By-products Industry*. 2006, National Renderers Association: Alexandria, VA.
[2] Orellana, J.L.; Smith, T.D.; Kitchens, C.L.; *Liquid and supercritical CO2 extraction of fat from rendered materials.* J. Supercritical Fluids, 2013. **79**(0): 55-61.
[3] Orellana, J. L.; Johnson, K. T.; Kitchens, C. L., *CO2 assisted mechanical expression of fat from rendered materials*. J. Supercritical Fluids 2014, **94** (0), 154-159.

Impacts and Significance: This research has potential for direct impact in the rendering process, with potential to enhance the continuous mechanical pressing of rendered materials with the addition of CO_2. The potential enhancements include the ability to obtain improved pressing yields with reduced torque or energy usage. Successful demonstration of this technology is of significant interest to many players across the rendering industry. For example, the HF German counterparts to Crown were very interested in our results. Feedback from highlights in Render Magazine has come from Renderers in Brazil and other companies including Green Recovery Technologies (http://greenrecoverytech.com). Results from this research are also of interest to the pet food industry who are interested in capabilities to obtain a low fat content poultry meal without the use of organic solvent extraction or excessive pressing that can result in material char.

Publications and Presentations: Publication of the pilot scale trial results is in preparation and is awaiting further process analysis and additional experimental trials to complete our parametric analysis. Prior publications and presentations related to this work include:

1. Orellana, J. L.; Johnson, K. T.; Kitchens, C. L., *CO2 assisted mechanical expression of fat from rendered materials*. J. Supercritical Fluids 2014, **94** (0), 154-159.
2. Orellana, J.L.; Smith, T.D.; Kitchens, C.L.; *Liquid and supercritical CO2 extraction of fat from rendered materials.* J. Supercritical Fluids, 2013. **79**(0): 55-61.
3. Orellana, J. L.; Johnston, K. T.; Kitchens, C.L. "*CO2 Assisted Mechanical Expression of Fat from Rendered Materials on the Laboratory and Pilot Plant Scale*", Proceedings of the AIChE Annual Meeting 2014, Atlanta, GA Nov. 16 – 21, 2014.
4. Orellana, J. L.; Kitchens, C.L. "*CO2 Assisted Mechanical Expression of Fat from Rendered Materials on the Laboratory and Pilot Plant Scale*", Proceedings of the AIChE Annual Meeting 2015, Salt Lake City, UT Nov. 9 – 13, 2015.

Outside funding: Two proposals were submitted to the USDA focused extracted rendered materials as organic fertilizers, specifically looking at measuring the nutrient release profiles and degradation mechanisms. Each proposal was budgeted just under $500k and neither was selected for funding.

Future Work: Future directions for this research will include further parametric studies of the poultry green crax pressing with the pilot scale system in order to determine the optimal operating conditions and full capabilities. We will also work with Crown to perform an economic analysis on the technology application.

Acknowledgments: **Tim Guzek, Brian Argo, and Tim Kedrowski (Plant Manager) of Sanimax who were critical in our ability to obtain the rendered materials for this study. We acknowledge Sanimax for the generous donation of significant amounts of roughly 12 tons of rendered materials for these studies. We acknowledge Chas Teeter and the Pilot Test Facility staff at Crown Iron Works for helping with the pilot scale trials. We acknowledge Mingzhe Jiang a doctoral candidate who helped with sample preparation and analysis. We acknowledge Dan Hildebrandt with Central Biproducts and Phil Anderson and Butch Fosdick with Darling International for joining us at Crown on the testing days.**