FINAL REPORT

August 30, 2013

ECONOMIC SEPARATION OF FAT COMPONENTS FROM RENDERED MATERIALS USING CARBON DIOXIDE

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Date Submitted:	February 8, 2012
Start Date:	July 1, 2012
Duration of Project :	12 months

Lay Summary:

The goal of this work is to use green, tunable solvents to process rendered materials into value-added co-products for energy, consumer products, commodity chemicals/materials, and an up-graded feed source for livestock, poultry, pets, and aquaculture. Current uses of rendered by-products are largely limited by the cost prohibitive separation into feed stocks of requisite purity and composition. We foresee opportunities for more selective separations that will preserve value added content within the rendered material, as well as, provide more efficient separations that could reduce energy costs. This research will focus on the use of tunable solvents to isolate value added fractions of the rendered material. The tunable solvent used for this work is supercritical and liquid carbon dioxide (CO_2) . CO_2 is referred to as tunable solvents because it's solvent properties can be altered by changing the temperature and pressure.

 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. Additionally, the separation of extracted fats from the CO_2 is achieved by simple depressurization or temperature swing, enabling facile CO_2 recycle. In this report our findings are primarily focused on the development of CO_2 -assisted mechanical expression (pressing) of fat from rendered materials. This technology has been demonstrated and implemented with seed oils but has not been applied to rendered materials until now.

Our results demonstrate that a low fat poultry meal (as low as 2.3% fat) can be achieved using 1) moderate CO₂ pressures that are achieved in industrial scale CO₂ extraction units and 2) less than 2 tons of force required by the press, which is on the order of the forces achievable in conventional screw presses. It is our impression that the value-added price of fats and an emerging market in low-fat meals for pet food and animal feeds will provide a market for this technology. Per the suggestion of the ACREC Industry Board Members, we have learned that Crown Iron Works does have a commercial unit marketed as High Pressure Liquid Extraction (HIPLEX). HIPLEX is a mechanical screw press designed to inject liquid CO₂ into the press to enhance the oil recovery from seeds at a scale of up to 500 tons per day. This technology is geared to seed oil recovery but may also have application in rendered materials. We are currently in discussions with Bruce MacKinnon and Chas Teeter with Crown Iron Works, as well as, Jeff Hendrix with The Dupps Company, and Dave Kaluzny with the Kaluzny Bros., Inc. We foresee that further developing these industry partnerships will lead to feasibility discussions and potential implementation of this technology.

Objective (s):

- 1. Liquid and supercritical CO₂ extraction of fats from poultry meal, meat and bone meal, feather meal, blood meal and DAF sludge using a lab-scale semi-batch extraction unit.
- 2. Thermodynamic modeling of the CO₂ Fat extraction which will aid in scale-up modeling and design of integrated operations.
- 3. Investigation of a CO₂-assisted mechanical pressing process in order to enhance the fat recovery during the pressing process.
- 4. Explore opportunities with industrial partners that include The Dupps Company, who designs, builds, and installs process equipment for the rendering and other industries.

Project Overview: This overview is planned for submission to the Journal of Supercritical Fluids for Peer-reviewed Publication.

Introduction

The rendering industry converts the by-products or inedible parts from the animals produced for human consumption into value-added products, commonly known as rendered materials (RM).¹ There are approximately 250 rendering facilities in North America that annually produce 18 billion pounds of RM per year, representing an important contribution to society and sustainability of the food industry.^{2,3} This industry is also green and environmentally beneficial, reducing the amount of waste while recycling carbon and energy into valuable feed ingredients and biofuels.³ The RM products consist of approximately 50% protein-based meals and 50% fats, which are commonly used in soap and personal care products or as a biofuel feedstock.³ The fat is separated from the protein matrix via screw press, which can leave 8 to 15% fat remaining in the protein meals.⁴ Thus, new methods for more efficient fat isolation are desired, especially since the fat is a higher value product.

Screw pressing offers the advantages of producing uncontaminated oil with low initial and operational costs, however, the low separation efficiencies in the current rendering process produces a material with 8-15% residual fat.¹ It is well know that extraction with organic solvents, such as hexane, yield higher efficiencies (>99%), but produces contaminated oil that requires refining.⁵ High pressure CO₂ is a greener alternative extraction solvent for a clean and selective separation of fats that has been widely used in the extraction of seeds, decaffeination of green coffee beans, and other areas in the food industry.⁶⁻⁹ Past research has shown that extraction of flaxseed oil with supercritical CO₂ yields approximately 28% more fat than screw pressing and just 9% less than hexane extraction.⁴ Liquid and supercritical CO₂ were also recently used for the extraction of fat from rendering materials, reducing the fat content to less than 1%.¹⁰ This process offers the advantages of separating a clean fat and the possibility of facile solvent recycling. However, the amount of CO₂ needed is relatively high due to the low fat solubilities ranging between 0.5 to 6.5 g/L depending on pressure and temperature.¹⁰

GAME (Gas-Assisted Mechanical Expression) is a process that combines mechanical pressures and the presence of CO₂ as a solvent to dissolve into oils and enhance the pressing operation. This enables higher fat recoveries using only a fraction of CO₂ compared to the aforementioned traditional CO₂ extraction. This is possible because the solubility of CO₂ in oils can be 50% higher than the solubility of oil in CO₂.^{11,12} This process was first introduced by Venter et al. for the separation of cocoa butter.¹³ In GAME, a gas-expanded liquid is formed by saturating CO_2 in the oil or fat, significantly reducing the viscosity of the mixture compared to pure lipids.¹⁴ This reduction of the viscosity allows for an increase in expression yield compared to conventional mechanical expression, since the lipids are drained more easily through the compressed bed.¹⁵ Moreover, this reduction in viscosity is also accompanied by a reduction of energy required for the separation process.¹⁶ Other possible factors affecting the enhanced separation are the freeing of oil due to disruption of the oil cell walls, and a reduction of interfacial tension of the oil.¹⁶ Additionally, it is possible that the undissolved CO₂, which is in equilibrium with the oil-CO₂ mixture, displaces the oil-CO₂ mixture contained in the filter cake.¹⁷ This behavior can be attributed to a higher density of the oil compared to supercritical CO₂, which actually increases when it is saturated with CO_2 .¹⁸

The aim of this work is to increase the fat extraction yield from rendered materials using CO_2 assisted mechanical expression. This method is expected to reduce the amount of CO_2 utilized compared to the liquid and supercritical CO_2 extractions in our semi-batch extraction results, as well as, reduce the energy requirement compared to conventional mechanical pressing. In this study, mechanical pressures between 300 and 2000 bars and CO_2 pressures between 69 and 241 bars were evaluated and compared to conventional expression. The effect of temperature (25, 40, 60 and 100°C) on the yield was also studied in this work.

Materials and Methods

Materials

Feed grade poultry by-product meal was used as the rendered material (RM) in our testing and was kindly donated by Valley Proteins Inc. The rendered material was used as received without any sample modification. The composition of the material was 12.1 wt.% fat and 7.0 wt.% moisture content. Carbon dioxide used in the experiments was purchased from Airgas and ACS grade n-hexane was purchased from VWR. A 10 ton hydraulic press (Torin[™] Big Red jack model #T51003) was purchased from Northern Tools. The extraction cell (piston, upper cylinder, lower cylinder, cylinder stand, and sieve plate) were designed in Solidworks and manufactured by Clemson Machining and Technical Services. Buna-nitrile O-ring with 2 backup rings was used on the piston assembly to seal the cylinder.

Experimental Set-up

The separation of fat from the rendered material was performed in batch mode using the designed high pressure cell with mechanical pressing capability, equipped with a hydraulic press as seen schematically in Figure 1. A Teledyne Isco 500HD syringe-pump connected to a recirculating heating bath was used to deliver the CO_2 at the desired pressure and temperature. A total amount of 5.0 g of RM was used in each experimental run. Heating tapes were wrapped around the top and lower cylinders were connected to an Omega CSC32 temperature controller in conjunction with a Payne Engineering 18TP variable voltage controller. K-type thermocouples provided feedback to the controllers. Before each experimental run, the cylinders were allowed to equilibrate at the specified temperature. The piston was lowered on top of the sample in each run to provide an initial compaction of the material.

High pressure CO_2 was introduced into the extraction cylinder for specific periods of time to reach equilibrium with the RM and fat. After this equilibrium time, the mechanical pressure was raised over the course of one minute to the desired force and held constant for a specific period of time. The expressed lipids passed through a 10 μ m frit filter and sieve plate to the collection chamber while the RM remained in the upper cylinder. The cylinder was then depressurized over the course of 1 min and the extracted fat was collected in a recovery flask filled with n-hexane. The extracted RM was collected and analyzed by the Agricultural Laboratory at Clemson University to determine the remaining fat content by a Soxhlet hexane extraction.

Experimental Procedure

The fat separation runs have two time-dependent parameters that can influence the yield of lipid expression. The equilibrium time is required before the mechanical pressure to allow the CO_2 to dissolve and equilibrate with the fat. The correct pressing time will allow a complete drainage of the fat- CO_2 mixture from the RM cake. To evaluate the equilibrium times on the extraction yield, the samples were allow to equilibrate for 0, 5, 10, 20, and 40 min at 40 °C and CO_2 pressure

of 172 bar before applying the mechanical pressure for the extraction for 10 min. In another set of experiments at a fixed equilibrium time, different pressing times in the same range (0 to 40 min) and conditions (40°C and CO₂ pressure of 172) as the previous experiment were evaluated. According to the results obtained, 20 min of equilibrium and 20 min of pressing were sufficient for equilibrium to be reached and used in the remaining experiments.

The effects of the effective mechanical pressure (P_{eff}) (70-1880 bar) and CO₂ pressure (0, 69, 103, 172, and 241 bar) on the extraction yield were studied at 40°C. P_{eff} is defined as the mechanical pressure exerted by hydraulic pump on the RM material minus the pressure of CO₂ inserted in the extraction cell. For these experiments, the CO₂ was introduced at the desired pressures and allowed to equilibrate before exerting specific forces (1, 2, 4.5, 6 US ton) that were converted to the P_{eff} in units of pressure (bars). The effect of temperature on the rendered fat extraction was studied at a constant mechanical force of 4.5 ton (1410 bar). These experiments were conducted at temperatures ranging between 25 to 100 °C using 103, 172, and 241 bars of CO₂ pressure.



Figure 1. Schematic diagram of the CO_2 assisted mechanical expression of rendered materials. (1) Hydraulic Ram; (2) Piston assembly; (3) Upper Cylinder; (4) Heating tape; (5) Rendered Material; (6) Sieve Plate and 10 μ m frit filter; (7) Lower Cylinder; (8) Collection Chamber; (9) Cylinder Stand; (10) Inlet CO2 valve; (11)Outlet CO2/lipid valve; (F) Mechanical Force gauge; (P) Pressure sensor; (T) Thermocouple.

Fat separation mechanisms

The displacement was measured using a Fisher Scientific Digital Caliper with a repeatability of 0.01 mm. This value was determined by measuring the distance that the bottom of the piston

would displace during the extraction process. The displacement experiments were performed at 40 °C at a CO₂ pressure of 103 MPa. The first measurement was taken immediately after reaching the desired mechanical pressure and subsequently every 1-5 minutes, assuring that the mechanical pressure was kept constant during the extraction.

The effects of pre-treating and post treating the RM sample with CO_2 were studied also at 40 °C. First, the RM sample was allowed to equilibrate with CO_2 at 103 bar for 20 min. The CO_2 was released from the cylinder and mechanical expression was applied in absence of CO_2 . For the post-treatment, the RM sample was first mechanically expressed for 20 min. The sample was removed and the pellet was pulverized and place again in the expression chamber for CO_2 extraction (103 bar) for 20 min with no mechanical pressure.

Results and discussion

Optimum experimental conditions

The necessary time to allow CO_2 to dissolve in the rendered materials was determined by experiments conducted at 40°C and CO_2 pressure of 172 bars. Equilibrium times up to 40 min demonstrated that at least 5 min are needed to reach equilibrium as observed in Figure 2. After 5 min, the equilibrium time does not influence the yields, where yield is defined as the fat extracted as a mass percentage of the original fat in the RM. An equilibrium time of 5 min is relatively low compared to other gas-assisted expressions of oilseeds which were reported to be higher than 30 min.⁵ Similar behavior was found in previous work, where CO_2 was able to easily penetrate the material and dissolve the fat, because the RM is finely ground with high porosities and particle sizes less than 100 μ m after extraction of fat.¹⁰

Figure 2 also shows the effect of pressing time on the yield, requiring a minimum of 20 min of pressing for a maximum extraction yield. Previous research report times of around 10 min for the extraction of vegetable oils.^{5,13} This behavior could be explained by the lower viscosities compared to animal fats, resulting in faster times for the fat to drain from the cake. For the purpose of this work, the extraction at different pressures and temperatures will be conducted for 20 min for both the equilibrium and pressing times.

Effect of CO₂ pressure and effective mechanical pressure

The influences of CO₂ pressure and P_{eff} on the expression yield are shown in Figure 3. The yields are observed to increases with CO₂ pressures, obtaining a highest value of 81% at CO₂ pressure of 241 bar and P_{eff} of 390 bar. On the other hand, the lowest extraction yields were obtained for the conventional expression which showed at best a 41.3% yield. The separations conducted at the lowest CO₂ pressure, 69 bar, had a 27% higher absolute yield than conventional expression with no CO₂, while increasing the CO₂ pressure from 69 to 241 bar only increased the yields by approximately 12%.

The trend of the extraction yields as a function of P_{eff} in Figure 3 exhibit a maximum value at around $P_{eff} = 500$ bar. This behavior also occurs with the conventional separation without the use of CO₂ and can be explained by understanding the variables involved in the drainage of the fat through the compressed cake. The superficial velocity of the fat (\hat{v}_F), or fat drainage, is described by the one dimensional version of the Darcy's law¹⁹ as follows:

$$\hat{v}_F = -\frac{B}{\eta_F} \frac{dP}{dz}$$
 Eq. 1

where B is permeability, η_F is the dynamic viscosity of the fluid, and dP/dz is the gradient of fluid pressure. As the mechanical pressure is exerted on the material, the oil pressure (the driving force) increases, while the permeability decreases, leading to a further increase of the oil pressure. However, at some point the permeability becomes so low that it disrupts the fat drainage and reduces the extraction yields.¹⁶ For the system in this work, this point seems to occur at some pressure around 630 bar for the conventional expression, and it may be the reason why some gas assisted expressions of oils reported in the literature are usually conducted at pressures not higher than 550 bar.^{5,13} This optimum pressure cannot be accurately determined from Figure 3, but it is in the range of P_{eff} = 600 bars.



Figure 2. Effects of the equilibrium time (\bullet) and pressing time (\bigcirc) on the extraction yields of rendered fats.

According to Darcy's equation, a reduction of the viscosity increases the drainage of the fat. The dissolution of CO_2 in oils has been shown to reduce the viscosity to 10% of the original oil viscosity at a CO_2 pressure of 150 bar.²⁰ However, other studies have shown that above ~150 bar the viscosity of the oil- CO_2 mixture does not vary significantly.^{14,20} This can explain why in Figure 3, the extraction yields between 172 and 241 bar are essentially the same. Hence, it is not advantageous to conduct the RM pressing at CO_2 pressures higher than 150 bar or P_{eff} greater than 600 bar. Figure 4 also presents the expression yield at CO_2 pressure of 138 bar and 40 °C with more experimental points in the lower range of P_{eff}, showing the maximum yield at around 550 bar.



Figure 3. Expression yield curves at different CO₂ pressures and 40°C as a function of effective mechanical pressure. (a) Extraction yields and (b) fat contents after extraction. Δ , 0 bar; \blacklozenge , 69 bar; \Box , 103 bar; \blacktriangle , 172 bar; \bigcirc , 241 bar.



Figure 4. Expression yield curve at CO₂ pressure of 138 bar and 40 °C.

Temperature effect

The effect of temperature on the expression yield was evaluated at pressures of 103, 172, and 241 bar (Figure 5). For the lowest pressure, the temperature was observed to have a more pronounced influence, increasing the yield by 20% going from 25°C to 40°C. However, the overall trend suggests that temperature slightly reduces the extraction yield as observed in Figure 5. This behavior can be explained by the reduced solubility of supercritical CO_2 in liquids as temperature is increased when the pressure is below a determined critical value.¹⁶

Expression Mechanisms

The reduction of viscosity of the fat as a result of the dissolution of CO_2 can be one of the main reasons for the higher extraction yields for the GAME process. The viscosity reduction should be reflected by the displacement of the piston along the extraction time. The displacement versus time

is presented in Figure 6 for conventional expression and GAME at a P_{eff} of 628 bar and 40 °C. It can be observed that the displacement for GAME is significantly higher than for the conventional expression, indicating a significant reduction of the viscosity upon de addition of supercritical CO₂ and drainage of the mixture. Although this result was expected because of the reduced viscosities of fat-CO2 mixtures, this mechanism could have not been the rate-limiting step in the separation due to more prominent effects resulting from the other mechanisms. These mechanisms are the rupture of cell walls by swelling due to CO2 dissolved in the oil, entrainment of fat by CO2 during depressurization, and displacement of oil by dissolved CO2.



Figure 5. Temperature effect on the extraction yield at different pressures. \blacklozenge , 103 bar; \Box , 172 bar; \blacklozenge , 241 bar.



Figure 6. Displacement of piston during extraction at constant mechanical pressure (Peff=628 bar) and 40 °C. GAME expression (triangles) and conventional expression (squares)

The rupture and entrainment mechanisms were evaluated by pre-treating and post-treating the RM sample with CO₂, respectively. For the former, the sample is first treated with CO₂ for the same time than GAME but with no mechanical pressure in order to break the cell walls of the RM. The CO2 is then released and mechanical pressure is applied for a conventional expression. The final fat percentage in the RM is estimated, and a different between the conventional expression and pre-treated sample will be as a result of the rupture of the cell walls. For the entrainment experiment, CO2 was equilibrated with the sample with no mechanical pressure after conventional expression for the same amount of time than GAME. Any difference in the final fat content between the post-treated sample and the conventional expression can be attributed to the entrainment of fat in the CO2 upon depressurization. As it can be observed in Figure 7, there is no difference between the entrainment and conventional expression, indicating that this mechanism is not a rate-limiting step. On the other hand, the expression yield for the rupture experiments is about three times higher than for conventional expression, indicating a significant cell rupture due to the dissolution of CO2 in the fat. This result agrees with the effect of the equilibrium time on the yield as discuss earlier, where at least 5 min of equilibrium time were needed for a maximum extraction yield. As shown in Figure 7, the GAME extraction yield is still significantly higher than the rupture experiment. This effect can be attributed to the reduction of viscosity of the fat-CO2 mixture, which increases the fat drainage though the RM and thus the expression yield.

Finally the last mechanism, the displacement of oil by CO2, may not affect significantly as it can also be observed from Figure 6, where the final displacement is significantly higher for GAME than for conventional extraction. This result indicates that at the end of the extraction at the same effective pressure, the total volume is much less for the GAME method, indicating that both, CO2 and oil migrated out of the cake. In a different case, if the final displacement were the same for both methods, the CO2 would then replace the volume of the oil, showing that the displacement mechanism was significant.



Figure 7. Yields of conventional, rupture, entrainment and GAME experiments for rendered materials.

Conclusions

CO₂ assisted mechanical expression of fats from rendered material was successfully conducted in this work, obtaining a highest yield of 81% compared to 41% achieved with conventional expression. This yield represents a reduction of the remaining fat content from the initial 12.1% to 2.3%. The minimum equilibrium time before the extraction was found to be 5 min, demonstrating a fast CO₂ dissolution into the fat component of the rendered material. An optimum effective mechanical pressure was observed at around 600 bar, which can be attributed to a reduced permeability of the CO₂-fat mixture through the RM matrix at high mechanical pressures, as described by Darcy's law. The CO₂ pressure increases the extraction yields; however, at pressures higher than 172 bar, the change becomes negligible possibly due to the viscosity of the oil-CO₂ mixture, which has been reported not to decrease significantly beyond such pressure.^{14,20} The two mechanisms identified governing the gas-assisted extraction were the reduced viscosity of the fat-CO₂ mixture, which increased the drainage of fat through the cake, and the rupture of cell walls due to swelling as a result of the CO₂ dissolution. Overall, it was demonstrated that high extraction yields can be achieved when conducting a gas-assisted extraction on RM, utilizing only a fraction of the amount of CO₂ used for CO₂-only extractions.

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Impacts and Significance: Based on this work, we have found that the optimal conditions for CO_2 -assisted mechanical expression of fat from rendered poultry meal is around 600 bar (8700 psi) of effective mechanical pressure, 172 bar (2500 psi) of CO_2 pressure, and 40°C. Our results demonstrate that at these conditions, the fat content could be reduced to as low as 2.3% fat. These results translate to a low-fat meal using 1) moderate CO_2 pressures that are achieved in industrial scale CO_2 extraction units and 2) less than 2 tons of force required by the press, which is on the order of the forces achievable in conventional screw presses.

It is our impression that the value-added price of fats and an emerging market in low-fat meals for pet food and animal feeds will provide a market for this technology. Per the suggestion of the ACREC Industry Board Members, we have learned that Crown Iron Works does have a commercial unit marketed 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 at a scale of up to 500 tons per day. This technology is geared to seed oil recovery but may also have application in rendered materials. We plan to continue pursuing industry partnership for feasibility discussions and potential implementation of this technology.

Publications:

• J.L. Orellana, Advanced Biomaterials from Renewable Resources: an Investigation of Cellulose Nanocrystal Composites and CO₂ Extraction of Rendered Materials, Ph.D. Dissertation, (August 2013).

- J.L. Orellana, K.T. Johnson, C.L. Kitchens; CO₂ Assisted Mechanical Expression of Fat from Poultry Meal; To be submitted to *J. Supercritical Fluids*, (Sept. 2013).
- Orellana, J. L.; Smith, T. D.; Kitchens, C. L.; Liquid and Supercritical CO2 Extraction of Fat from Rendered Materials. *J. Supercritical Fluids* **79**, 55-61, (2013)
- Orellana, J. L., Smith, T., Kitchens, C. L., "Liquid and Supercritical CO2 Extraction of Fat from Rendered Materials and Solubility Correlation" *Proceedings of the 10th International Symposium on Supercritical Fluids*. San Francisco, CA (May 2012).

Presentations:

- Orellana, J. L., Kitchens, C. L., "Liquid and Supercritical CO2 Extraction of Fat From Rendered Materials", Proceedings of the AIChE Annual Meeting 2012, Pittsburgh, PA Oct. 28 – Nov. 2, 2012.
- Orellana, J. L., Smith, T., Kitchens, C. L., "Liquid and Supercritical CO2 Extraction of Fat from Rendered Materials and Solubility Correlation" 10th International Symposium on Supercritical Fluids. San Francisco, CA, May 13th-16th 2012. (Poster).
- Kitchens, C. L. "Extraction of Fat from Rendered Material using Tunable Fluids" Presentation at the National Renderers Association Annual Meeting in Tucson, AZ, October 18th, 2011.
- Orellana, J.L.; Kitchens, C.L. Supercritical and Liquid CO2 Extraction of Fat from Rendered Materials. Presentation at SACNAS 2011 National Conference, San Jose, CA, October 2011.

Outside funding:

"Enhanced Nitrogen Use Efficiency through a Protein Byproduct Delivery Mechanism"; Submitted to the Virtual Fertilizer Research Consortium; Award Amount 655,766; 9/1/12 - 8/31/14.

Future Work: The future work for this project will likely lie with industrial outreach to demonstrate the feasibility of large-scale CO_2 -assisted mechanical expression of fats from rendered materials. The batch press experiments performed in this work do demonstrate feasibility and provide a basis for understanding the mechanisms of fat expression; particularly understanding the effects of mechanical pressure, CO_2 partial pressure, and temperature. One significant difference may be that the CO_2 -assisted mechanical expression will be applied to the crax material rather than the finished rendered material.

We plan to further explore opportunities with industrial partners that will include discussions and potential site visits with:

- Jeff Hendrix with The Dupps Company: We have been trying to schedule a visit over the past year. Two separate visits have been planned and scheduled and then canceled at the last minute.
- Bruce MacKinnon and Chas Teeter with Crown Iron Works: In 2004, Crown Iron Works acquired the High Pressure Liquid Extraction (HIPLEX) technology from Harburg-Freudenberger (HF). The HIPLEX system is a mechanical screw press designed to inject liquid CO₂ into the press to enhance the oil recovery from seeds. According to MacKinnon, they tried using the HIPLEX system with rendered materials several years ago and achieved approximately 2% improvement. I am working on getting more details

of this test to see what the material and operating conditions were. I foresee that we can provide insight into the optimum operation conditions for the HIPLEX system based on our results.

- Dave Kaluzny with the Kaluzny Bros., Inc: I have been in touch with Dave Kaluzny II about the potential of using CO₂ technologies in their operations. Currently they use CO₂ for cooling and may have interest in our technology. I have provided him with literature and am waiting to hear if they would like to discuss options.
- SCAMEX is a French company (<u>www.scamex.fr</u>) that has designed and constructed pilot scale screw presses thaqt are capable of operating with CO2 partial pressure.

Acknowledgments: We acknowledge the Animal Co-products Research and Education Center (ACREC) at Clemson University and the Fats & Proteins Research Foundation for funding and guidance in this work; and Carolina By-Products and Valley Proteins in Ward, SC for the donation of rendered poultry meal.