

FINAL REPORT
September 17, 2012

**TUNABLE FLUIDS FOR ECONOMIC SEPARATION OF RENDERED MATERIALS
FOR FUELS AND POLYMERS**

**ECONOMIC SEPARATION OF FAT COMPONENTS FROM RENDERED
MATERIALS USING CARBON DIOXIDE**

Principal Investigator(s): Christopher Kitchens, Associate Professor
ckitch@clermson.edu
Department of Chemical and Biomolecular Engineering
127 Earle Hall
Clemson University
Clemson, SC 29634
(864) 656-2131/(864) 656-0784 FAX

Collaborators: Jose Orellana, Ph. D. Candidate
joseo@clermson.edu
Department of Chemical and Biomolecular Engineering
127 Earle Hall
Clemson University
Clemson, SC 29634
(864) 656-2131/(864) 656-0784 FAX

Date Submitted: March 17, 2010

Start Date: July 1, 2010

Duration of Project: 24 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. 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. Within this scenario, there are 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. These tunable solvents consist of supercritical and liquid carbon dioxide (CO₂) and CO₂ plus methanol co-solvent (a gas expanded liquid, GXL). These solvents are referred to as tunable solvents because their solvent properties can be altered by changing their temperature and pressure.

CO₂ is an attractive extraction solvent because it is non-toxic, chemically inert, inexpensive, abundant, and FDA approved. From a processing and separations standpoint, CO₂ is attractive because the solvent properties can be tailored by controlling the temperature and pressure. For example, the different components of the fat in rendered materials will have different solubility in CO₂ at different pressures. Additionally, the separation of extracted fats from the CO₂ is achieved by simple depressurization or temperature swing, enabling facile CO₂ recycle.

Objectives:

1. Extraction of poultry meal and crax with liquid and supercritical CO₂ in a semi-batch extraction unit that will enable characterization of the triglyceride, lipid, cholesterol, and other fatty acid fractions as they are extracted.
2. Measurement of the cloud points of the different fatty acid components in liquid and supercritical CO₂. This data will be compared to literature and used in an Aspen simulation of the extraction.
3. Process MBM with CO₂ and methanol mixtures. Methanol will increase the overall solvent strength and form in-situ methyl carbonic acid which will help to break up the protein and fat matrix. CO₂ also enhances the transport properties, providing gas-like diffusivities, which enable the solvent to penetrate the MBM matrix and enhance extraction.
4. Model the extraction in Aspen Plus and perform an economic analysis of the process. This modeling will use the experimental data we obtain and can be compared to hexane extraction.

Project Overview:

Introduction:

Rendered materials (RM) are produced by the rendering industry from the inedible fractions of animals produced for human consumption, which constitutes one third to one half of the total animal mass[1]. In 2009, the U.S produced 33 million cattle, 113 million hogs, 245 million turkeys and 8.6 billion chickens for human consumption. The animal by-products were processed in the 250 rendering facilities in North America and produced around 18 billion pounds of RM [2, 3]. Out of the total production, 52% are a combination of fats and greases; and the remaining 48% are

protein meals composed of meat-and-bone meal, poultry meal and feather meals [3]. Fats are non-polar soluble biomolecules consisting of triglycerides and fatty acids that unlike oils are solid at room temperature due to the high content of saturated fatty acids. Approximately 85% of all RMs, including a fraction of the fats, are produced for animal feed ingredients. The rest is used in a diversity of industries with nearly 3,000 applications identified [1]. A large fraction of the fat not used for animal feed is used in the manufacture of soaps and personal care products; however, since 2010 the biofuels industry has shown record production and has placed a significant demand on the fat from the rendering industry, more than doubling the amount of rendered fats used for the biodiesel production [4]. The processing of the inedible raw materials and the use of the by-products make an important economic contribution to this industry and also contributes to environmental and public health since rendering offers a more sustainable solution and a lower carbon footprint than other disposal methods [5].

The rendering process involves the application of heat, the extraction of moisture and the separation of fat. First the raw materials are ground to a consistent size and cooked with steam at temperatures from 115°C to 145°C for 40 to 90 min [2]. Moisture is boiled off and the free-fat is drained. The fat associated with the solids is mechanically removed by screw presses and the moisture associated with extracted fat is separated using centrifuges. The two main product streams of this process are the fats (greases, tallow, lard and poultry fat); and the protein meals which can contain 8-15% residual fat. Current market trends have resulted in elevated prices for the fat fractions to about \$0.46 per pound in 2011 for the inedible fats which represents a 96% increase since 2009 and a 330% increase since 2001 [3, 4]. Thus, it is desirable to find alternative methods that are sustainable and economically viable for a fat selective extraction of rendered materials.

Mechanical extraction offers quite low initial and operational costs and produces uncontaminated oil; yet it yields relatively low extraction efficiencies, which may or may not be desired depending on the end product. Solvent extraction with organic solvents is a high efficiency extraction (>99%) but produces low quality oil that requires refining [6]. An alternative solvent that has attracted considerable attention for fat extraction is liquid or supercritical carbon dioxide (LCO₂ or SCCO₂). It has been shown that SCCO₂ extraction of flaxseed oil yields about 28% more fat than screw expression and just 9% less than hexane extraction [7]. CO₂ offers the advantages of ease of complete separation of the fat with no residual solvent in the matrix or the lipids, and the potential for CO₂ recycling. CO₂ is a non-toxic, non-flammable and relatively inexpensive solvent that has been used for a wide variety of applications that include separations, reactions and materials processing [8, 9]. Supercritical CO₂ can be advantageous over liquid CO₂ for extractions from certain matrices when mass transfer limitations exist, because the density is on the order of a liquid but the viscosity and diffusivity are on the order of a gas. Nevertheless, for many supercritical extraction systems a retrograde solubility phenomenon may occur, where decreased solubility of the solute occurs at elevated temperatures. This phenomena occurs below a pressure referred as the “cross-over” point and it is the result of reduced solvent strength as a result of reduced density in the high compressibility region of the fluid (i.e. close to the critical point). The density effect on solubility is not compensated by the increased solute volatility with increasing temperature [10]. This effect results in the fat having a higher solubility at lower temperatures in liquid CO₂ as compared to supercritical CO₂ at an equivalent pressure. This affords the advantage of using LCO₂ at lower temperatures and pressures for the extractions, which are beneficial for capital and operating costs, as well as, the recovery of volatile and thermally labile components [11].

The use of supercritical fluids in the food industry is widely established [9, 12, 13]. The first commercial supercritical extraction was performed in 1978 by Hag A.G for the decaffeination of green coffee beans [10]. Commercial applications at present include decaffeination of coffee and tea, extraction of natural colors, natural flavorings, antioxidants, nutraceuticals, and hops, as well as, extraction of lipids and cholesterol from egg yolks, milk fat, beef and pork [9]. The supercritical extraction of lipids for the production of biodiesel is also a promising and expanding research area [14, 15]. Extraction of specialty oils has received a great deal of interest due to the expanding demand for bioactive lipid components and the capability of CO₂ to preserve the flavors and aromas [16]. This is the primary reason LCO₂ is commonly used in the extraction of flowers [17]. In this work, LCO₂ and SCCO₂ were investigated in a continuous flow configuration for the extraction of the residual fat from rendered poultry meal proceeding from the last stage of the rendering plant process. The effect of the temperature (25 °C, 40°C and 50 °C), pressure (69 to 345 bar), flow rate (5 to 25 mL/min) and mass of CO₂ on the extraction yield and the fat solubility were investigated. The composition of fatty acids before and after the extraction was analyzed by gas chromatography. Solubility data was estimated from the extraction curves and correlated as a function of temperature and density using the Chrastil model. The maximum extraction yields and solubilities of the fat in LCO₂ and SCCO₂ were compared. LCO₂ was found to be more effective for the extraction of fat at the conditions studied in this work.

Materials & Methods:

Poultry meal was used as the rendered material which was kindly donated by Carolina By-Products and Valley Proteins in Ward, South Carolina and was used as provided with no further sample preparation. The composition of this material was 14.2 ± 0.2 wt% fat and an approximate content of 7% moisture and 63% crude proteins. The size of agglomerated particles of the poultry meal was 100-300 μm . ACS grade n-hexane was purchased from VWR and industrial grade Carbon Dioxide was purchased from Airgas.

Continuous Flow Extraction System Description

The apparatus used for the continuous extraction of fat from rendered materials is shown schematically in Figure 1. A Teledyne Isco 500HD syringe pump connected to a heating bath was used to deliver the CO₂ at a desired temperature and pressure while monitoring the volumetric flow rate. A heating column placed before the extraction column, was used when the extraction was performed at supercritical conditions in order to maintain the CO₂ at the desired temperatures of extraction. The extraction column consisted of 1/2" stainless steel tubing with metal frits with 10 micron porosity at both ends of the column which had an internal volume for approximately 4 grams of RM. The CO₂ flow rate was controlled using a heated back pressure regulator at the end of the system and the temperature was monitored using multiple K-type thermocouples and manually controlled with variable transformers. Pressure was monitored with a pressure transducer and indicator connected to the outlet of the extraction tube. A recovery flask was filled with hexane to collect the fat at the end of the system. The extracted RM was sent to the Agricultural Laboratory at Clemson University to determine the remaining fat by a Soxlet hexane extraction.

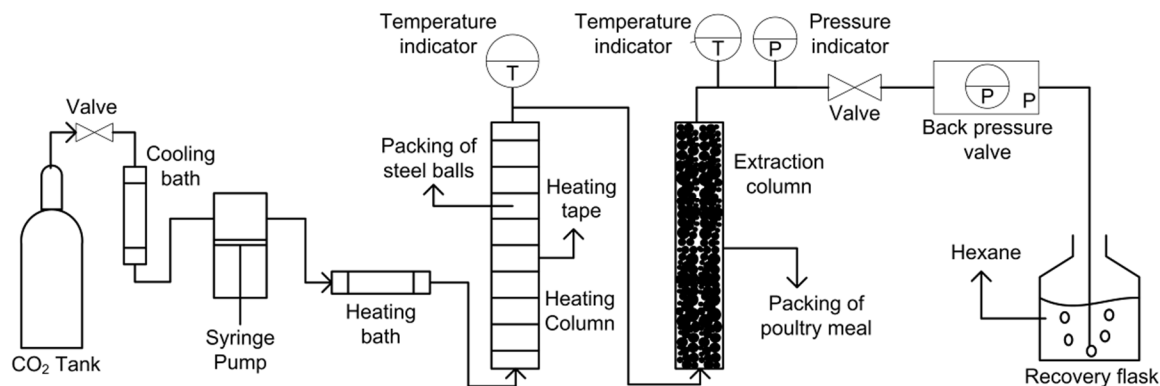


Figure 1. Schematic diagram of the continuous liquid and supercritical extraction.

LCO₂ and SCCO₂ Extractions of Rendered Fats

The extractions were performed at 25°C for LCO₂ and at 40°C and 50°C for SCCO₂ at pressures ranging from 69 to 345 bar. For SCCO₂, pressures below 210 bar required an exceedingly large amount of CO₂ in order to complete the extraction and therefore they were not performed. The effect of the flow rates (5 to 25 mL/min) on the extraction yield were also investigated for LCO₂. The density of CO₂ at each temperature and pressure was obtained from the National Institute of Standards and Technology (NIST) [18]. The solubility data was correlated as a function of density and temperature using the Chrastil model [19].

Gas Chromatography Analysis

The fatty acid composition of the fat before and after the extraction with LCO₂ and SCCO₂ was determined by an Agilent 5975C Series GC/MSD with a Flame Ionization Detector (FID) and using a GLC-90 column. First, the fats were methyl esterified with the following procedure: 20 µL of fat sample was reacted for 90 min at 70°C with 700 µL of KOH 10N and 6.30 mL of methanol. The same was performed with 60 µL of methylation blank sample of myristic acid. Samples were cooled down and reacted with 700 µL of H₂SO₄ 24N for 60 min at 70°C. After the reaction, the samples were mixed with 4.5 mL of hexane and subsequently centrifuged at 1100g for 5 min. The supernatant was 20 times diluted in hexane for the GC injection. The volume of injected sample was 1 µL.

Results & Discussion:

The continuous extraction of fat from rendered poultry meal with LCO₂ and SCCO₂ at temperatures (25°C, 40°C and 50°C) and pressures (69 to 345 bar), gave maximum extraction yields ranging from 87.3% to 96.5% (Table 1); where the extraction yield is defined as the fat extracted as a mass percentage of the original fat in the RM. These extraction yields did not show any dependence with pressure or temperature. On the other hand, the extraction efficiency defined herein as the extraction yield obtained per gram of CO₂ used, increased with pressure but decreased with temperature. Thus, the minimum amount of CO₂ required for complete extraction among all the conditions tested occurred for LCO₂ at the highest pressure (Figure 2a), hence the highest extraction efficiency. For the complete extraction, the lowest fat content was 1.0±0.3 wt% for all the conditions tested as shown in Figure 2.

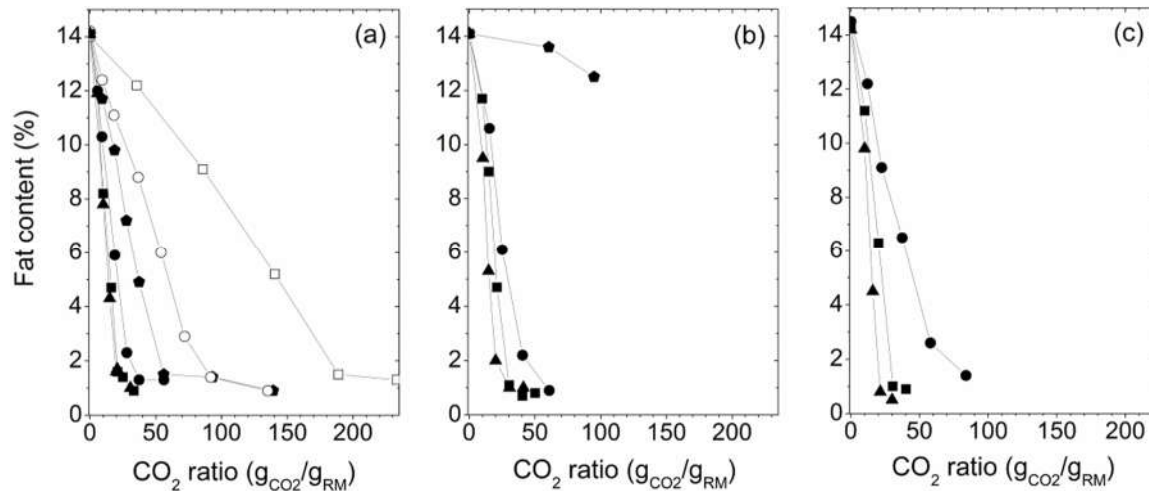


Figure 2. Fat content in rendered poultry meal after extraction as a function of the amount of CO₂ used in the extraction. (a) Liquid CO₂ at 25°C; (b) Supercritical CO₂ at 40°C; and (c) Supercritical CO₂ at 50°C. □, 69 bar; ○, 103 bar; ◆, 138 bar; ●, 207 bar; ■, 276 bar; ▲, 345 bar.

Effect of temperature, pressure and flow rate on the extraction efficiency

LCO₂ and SCCO₂ extractions curves in Figure 2 present the amount of fat remaining in the rendered poultry meal after extraction as a function of the CO₂ used at different temperatures and pressures. The extraction curves show the three regions usually found in natural product extractions: constant extraction rate period (first part) which is governed by the equilibrium solubility of the fat in CO₂; the falling rate period (transition); and the diffusion-controlled rate period which is observed in some of the curves at the end of the run. The slope of the lines observed in the constant extraction period increases with pressure due to an increase of CO₂ solvation power. Although not observed clearly in Figure 2, these slopes also increase when decreasing temperature. The solvation power of the CO₂ depends mainly on the CO₂ density and the volatility of the solute (fat), which are directly impacted by pressure and temperature. Due to the high compressibility of CO₂ in the vicinity of the critical pressure, density is more dominant than solute volatility when the conditions are close to this region.

As mentioned above, pressure increases the solvent power of CO₂ due to the increase of density which it becomes more noticeable at pressures near the critical point. As an example, for LCO₂ between 103 and 70 bar (Figure 2a), the difference in the CO₂ ratios required for complete extraction is about 80; while for the extractions between 345 and 207 bar this value is only about 35. It can also be observed that as the pressure increases to 345 bar the slope of the curves become closer to each other for the 3 temperatures studied. Although it was not studied in this work, it is expected that they will converge at the “cross-over” pressure, which may occur in this system at around 400 bar.

The temperature effect on the solvation power of CO₂ has a competing effect caused by both, density and solute volatility. Increasing temperature under constant pressure will decrease density but increase volatility and vice versa. If the conditions are below the aforementioned “crossover” pressure, density will be more dominant over the solute volatility and thus decreasing solubility

with temperature. This effect is commonly termed as retrograde solubility phenomenon which occurs in the regions of high compressibility, which is the case for this work. Once the crossover pressure is reached, the change of density with pressure becomes smaller and the volatility becomes dominant.

Table 1. Solubilities, extraction yield and extraction efficiency of rendered fat from poultry meal using LCO₂ and SCCO₂

	T	P	CO ₂ density	Fat solubility	Solubility Std. Error	Extraction Yield	Extraction Efficiency, EF
	°C	bar	g/L	g/L	g/L	(%)	(%/gCO ₂)
LCO₂	25	69	738.2	0.491	0.020	90.8	0.126
		103	823.0	1.252	0.036	93.6	0.288
		138	865.0	1.973	0.074	93.6	0.432
		207	918.6	3.957	0.086	90.8	0.816
		276	955.6	5.651	0.082	93.6	1.120
		345	984.6	6.474	0.442	92.9	1.245
SCCO₂	40	207	846.0	2.563	0.229	93.6	0.574
		276	896.0	4.054	0.408	94.3	0.857
		345	932.4	5.694	0.622	92.9	1.157
	50	207	792.4	1.638	0.078	87.3	0.391
		276	854.1	3.717	0.323	93.6	0.824
		345	896.5	5.647	0.630	96.5	1.193

No significant effect on the extraction efficiency (EF) was observed with variation of CO₂ flow rates between 5 and 25 mL/min investigated in this work, as can be observed in Figure 3. The EF in Figure 3 is the fat extraction yield divided by the CO₂ mass used for the extraction. This result indicates that the extraction reaches the equilibrium solubility concentration and that the intraparticle diffusion resistance is negligible for the flow rates investigated. This explains the high extraction efficiency of the LCO₂ since the system is controlled by the solubility of the solute in CO₂ and not by the mass transfer resistance. If the intraparticle diffusion were more dominant, SCCO₂ may prove to be more effective for fat extraction. The gas-like diffusivity of the supercritical fluid allows easier access through the particle pores, as compared to liquid CO₂, which would lead to higher extraction yields for mass transfer limiting matrices. In this case, this penetrating power of SCCO₂ is not required and rather the solvent power provided by the higher density of the liquid is preferred. This is likely due to the particles size of the finely-ground rendered poultry meal since intraparticle diffusion resistance becomes smaller for small particles sizes because of the shorter diffusion paths [20]. The particles of rendered material also decrease their size significantly after the extraction from around 200 μm to about 20μm in average as observed in Figure 4. This can also be another influencing factor on the high extraction yields obtained since the particle size decreases as the extraction is carried out. It should be mentioned that the decreased particle size can lead to material handling issues.

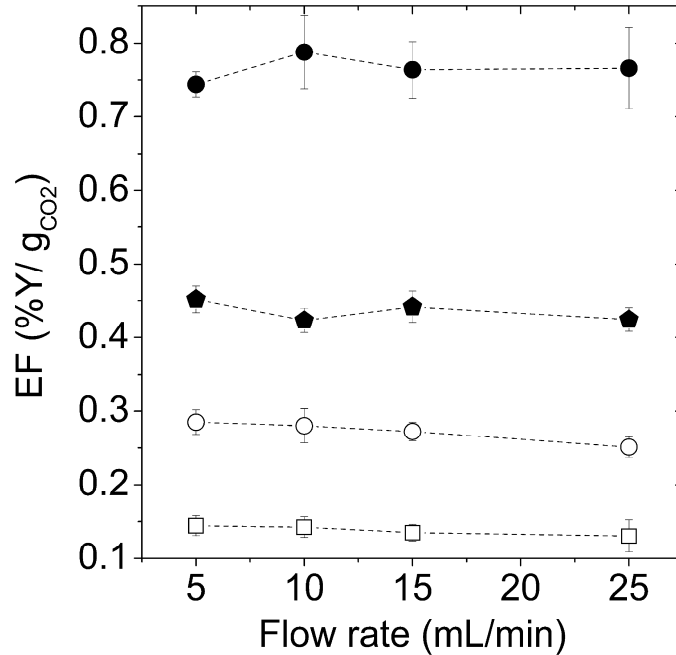


Figure 3. Extraction efficiency (EF) at different liquid CO₂ flow rates. □, 69 bar; ○, 103 bar; ◆, 138 bar; ●, 207 bar.

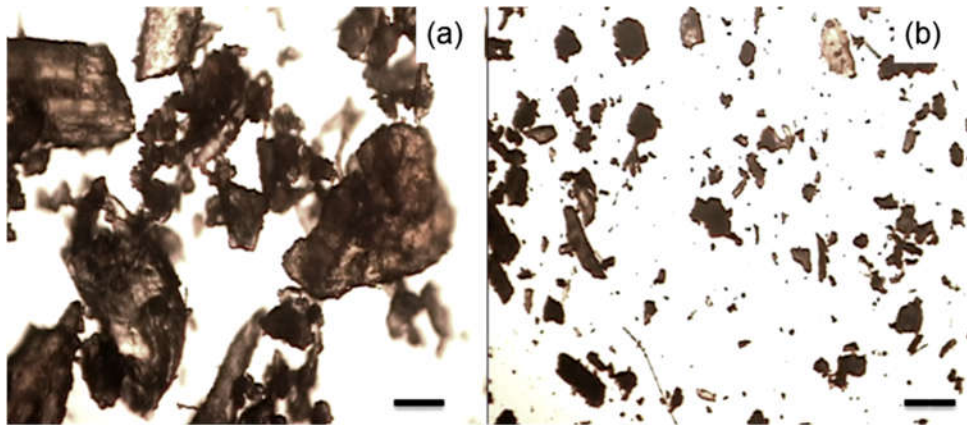


Figure 4. Optical Microscope image of RM. Scale bar: 100 μm . (a) Before the CO₂ extraction; and (b) After CO₂ extraction.

Solubility calculation and correlation with the Chrastil model

The fat solubility was determined from the linear trend observed in the extraction curves of Figure 2. This represents the extraction region that is governed by the solubility equilibrium of the components as proved in the flow rate experiments. The slope of the extraction curve (fat extracted versus amount of CO₂) can be defined as the solubility of the fat in the solvent, c (g/L); and calculated as described by Reverchon [21]. The equation is modified in this work to be applied to the axis plotted in Figure 2 as follow:

$$c = \frac{m_e}{V_s} = \frac{Y}{\left(\frac{V_s}{m_o}\right)} = \frac{-M * d_{CO_2}}{100} \quad \text{Eq. 1}$$

Where m_e is the mass of fat extracted, V_s is the volume of CO₂ used in the extraction, Y is the extraction yield, m_o is the initial mass of fat in the RM, d is the CO₂ density and M is the slope of the extraction curve. M was obtained by a linear regression of each extraction curve using the OrigenPro7 software; and d from the NIST Chemistry WebBook [18]. The solubility data and the CO₂ densities are presented in Table 1. As discussed before, the solubility of fat is higher for LCO₂ than for SCCO₂ due to the retrograde phenomena which makes solubility decrease with temperature as observed in Figure 5a. As the pressure reaches 345 bar, the solubility isotherms get closer to each other and will likely converge to the “cross-over” pressure. Figure 5b shows the single effect of the fat volatility on the solubility in CO₂, where as discussed earlier, at constant density the fat solubility in CO₂ increases with increasing temperature.

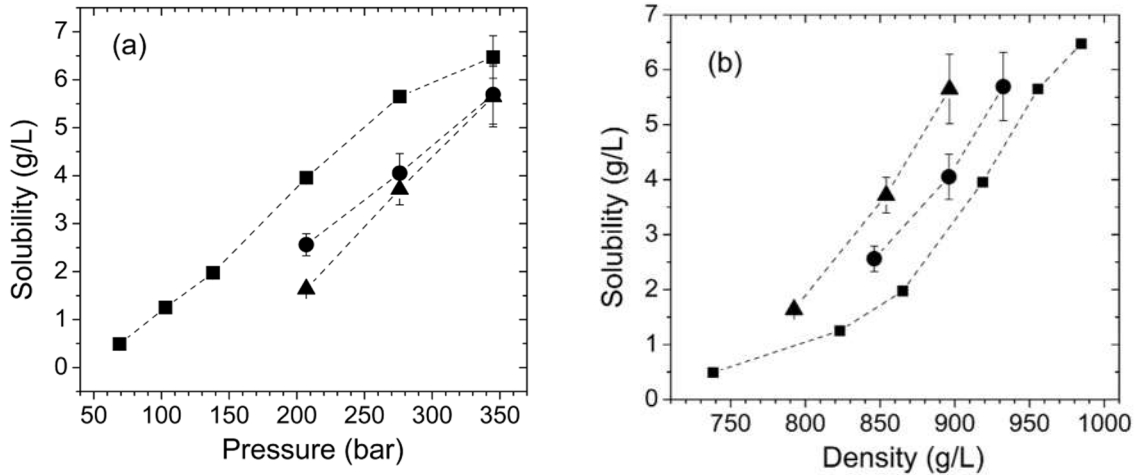


Figure 2. Solubility dependence of (a) pressure and (b) density. ■, 25 °C; ●, 40 °C; ▲, 50 °C.

The Chrastil model [19] was used to correlate the solubility of the fat as a function of the CO₂ density and temperature. The model is based on the hypothesis that one molecule of solute A (fat component) can be associated with k molecules of solvent B (CO₂). This association forms a complex molecule AB _{k} that it is in equilibrium. The Chrastil equation is given as follow:

$$\ln c = k \ln d + \frac{a}{T} + b \quad \text{Eq. 2}$$

Where k , a and b are constants to be determined by fitting the data. The constant k accounts for the solvation, which is the slope of the solubility isotherm and reflects the density dependence of the solubility. The constant, a , represents the heat of solvation and heat of vaporization of the solute and is also a measure of the temperature dependence of the solubility at constant density. Parameter b is dependent on the solute and solvent molecular weights and the association constant. A nonlinear regression was performed on the fat solubility data using Polymath 5.1 obtaining the following equation:

$$\ln c = 9.272 \ln d - \frac{2417.4}{T} - 53.86 \quad \text{Eq. 3}$$

The solubility prediction can be observed in Figure 6, which displays the estimated natural logarithms of the solubility versus density. The correlation accuracy was evaluated with the average absolute relative deviation (AARD) obtaining a 5.56% under the conditions studied in this investigation.

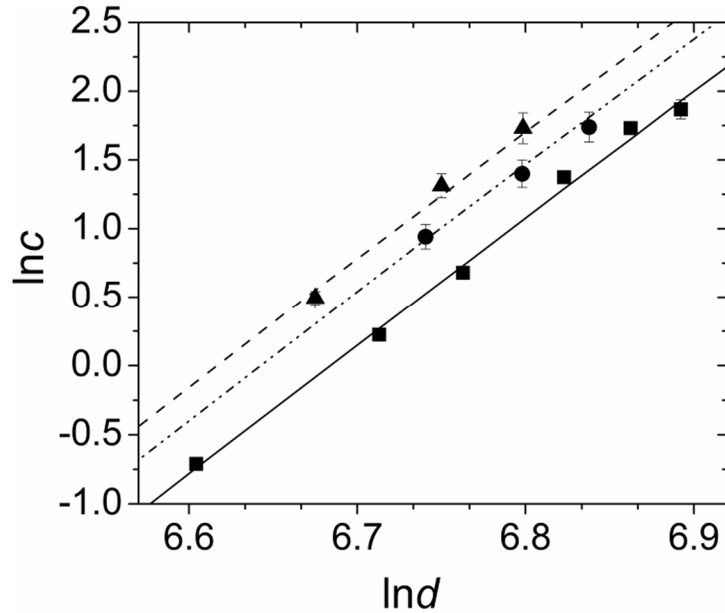


Figure 6. Plot of $\ln c$ vs $\ln d$ using Chrastil model for the experimental data at pressures between 69 and 345 bar and temperatures of: ■, 25 °C; ●, 40 °C; ▲, 50 °C. Lines represent the results from the Chrastil model: —, 25 °C; - - - 40 °C; - · - ·, 50 °C.

The modified Chrastil equation proposed by Sun and Li [22] was also evaluated to correlate the solubility in order to improve the results over the simple Chrastil model. This modified Chrastil equation is as follows:

$$\ln c = (k + k_1 \cdot d) \ln d + \frac{(a + a_1 \cdot d)}{T} + b \quad \text{Eq. 4}$$

This modification includes the additional parameters k_1 and a_1 to account for any nonlinear correlation with CO_2 density. The constants were estimated but the AARD obtained was only 5.58%, which it shows no improvement over the simple Chrastil equation shown above.

Fatty acid composition

Analysis of the fatty acid compositions from the rendered poultry meal before and after CO_2 extraction was conducted using gas chromatography. A total of 6 fatty acids were identified with a higher proportion of unsaturated fats than saturated as observed in Table 2. Most of the fatty acids present in the poultry fat [2] were identified in the GC analysis except for very small fractions of myristic (C14:0), margaric (C17:0) and linoleic (C18:3) acids. The fraction of lauric acid (C12:0) observed in the analysis was significantly higher than the reported in the literature. The difference between the fatty acid fractions observed before and after the CO_2 extraction is not very significant. However, it is worth noting that the concentration of unsaturated fatty acids decreased after the extraction indicating a possible higher solubility for unsaturated fats as shown

in the literature [23]. It was expected that about 7% of the total fat would correspond to a specific fatty acid that will not be present after the extraction because of the 1% of fat remaining in the rendered poultry meal. Nevertheless, it could be possible that the residual 1% is inaccessible fat in the poultry meal matrix or some other component that is hexane soluble but CO₂ insoluble. At the end of the linear trend on the extraction curves, the extraction is no longer controlled by the solubility of the fat but very likely by a diffusion effect where the intraparticle diffusion resistance is very significant. The extraction of the remaining 1% at this point may not be worth performing since it would require high amounts of CO₂, longer extraction times or higher temperatures above the crossover pressure

Table 2. Fatty acid mass fraction of rendered fats before and after extraction

Compound	Wt. %	
	Before Extraction	Extracted by CO ₂
Lauric (C12:0)	11.4	11.6
Palmitic (C16:0)	23.0	23.7
Palmitoleic (C16:1)	5.2	4.3
Stearic (C18:0)	6.6	9.9
Oleic (C18:1)	34.7	34.3
Linoleic (C18:2)	17.3	14.5
Unknown	1.8	1.8
Saturated	41.0	45.1
Unsaturated	57.3	53.0

Conclusions

LCO₂ and SCCO₂ were used for the extraction of fat from rendered poultry meal at different pressures (69-345 bars) and temperatures (25°C, 40°C and 50°C), obtaining maximum extraction yields between 87% and 97% for all the conditions. The starting material had a fat content of 14.2±0.2% which is residual from the mechanical press and cooking in the rendering plant from which the material was obtained. After the extractions, the fat content in the samples was reduced to a minimum value of 1.0±0.1% demonstrating the ability of the CO₂ to efficiently extract the fat from rendering materials. The fat analysis before and after the extraction yielded a total of 6 fatty acids, which were identified in agreement with the literature. It was observed that the fat composition before and after the extraction was not significant and that the residual fat content in the rendered poultry meal could be inaccessible fat in the matrix of the material.

Solubilities of the fat in CO₂ were calculated from the slope of the extraction curves at all the conditions tested. This estimation was possible because at the conditions of the extractions, the system reached the equilibrium solubility as proved by the flow rate experiments. These solubilities ranged from 0.491g/L to 6.474 g/L for LCO₂ at 69 bar and 345 bar psi respectively; and the solubility in SCCO₂ were intermittent between these values. It was observed that SCCO₂ at all pressures had lower solubilities than LCO₂ at the same pressures due to the retrograde solubility phenomena. The solubilities were successfully correlated with the Chrastil model obtaining an AARD of 5.56%. It was shown in this investigation that LCO₂ is more efficient than

SCCO₂ in the extraction of fat since the maximum extraction yields were the same for both fluids but less CO₂ is required for the liquid phase due to the higher solubility.

Impacts and Significance:

This work demonstrates the feasibility of fat extraction from rendered poultry meal. LCO₂ was proven to be more effective than SCCO₂ at low/moderate pressures. Moreover, nearly complete extraction was observed with all conditions tested. This process has potential to provide economic benefit based on the current market prices for fat components. Process modeling and economic analysis will further reveal this potential and the potential margins. This technology is applicable to lower value rendered materials as well as integration into traditional fat separation operations, specifically a CO₂ assisted mechanical pressing for enhanced fat expression. An invention disclosure on this technology was filed, which included an exhaustive literature and patent search.

Publications & Presentations:

- J. L. Orellana, T. D. Smith, C. L. Kitchens; Liquid and Supercritical CO₂ Extraction of Fat from Rendered Materials; Submitted to J. Supercrit. Fluids, June 2012.
- 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:

- Completion of the process modeling and economic analysis.
- Investigation of methanol as a co-solvent to enhance fat solubility during CO₂ extraction.
- Determine the quality and grade of the fat extracted using this process.
- Apply extraction to other material streams including meat and bone meal, feather meal, blood meal, and DAF sludge.
- Investigate the potential of CO₂ assisted fat expression during pressing.

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.

References:

- [1] D.L. Meeker, North American Rendering - processing high quality protein and fats for feed, Rev. Bras. Zootecn., 38 (2009) 432-U443.
- [2] D.L. Meeker, Essential Rendering - All About The Animal By-Products Industry, N.R. Association (Ed.), Arlington, 2006.

- [3] NRS, National Renderers Association, 2012. Available at: <http://nationalrenderers.org/>
- [4] K. Swisher, Market Report: Industry savors record prices and growing global demand, *Render Magazine*, 2012, pp. 10-18.
- [5] C.H. Gooding, Data for the Carbon Footprinting of Rendering Operations, *J. Ind. Ecol.*, 16 (2012) 223-230.
- [6] P. Willems, N.J.M. Kuipers, A.B. de Haan, Gas assisted mechanical expression of oilseeds: Influence of process parameters on oil yield, *J. Supercrit. Fluids*, 45 (2008) 298-305.
- [7] R.C. Pradhan, V. Meda, P.K. Rout, S. Naik, A.K. Dalai, Supercritical CO₂ extraction of fatty oil from flaxseed and comparison with screw press expression and solvent extraction processes, *Journal of Food Engineering*, 98 (2010) 393-397.
- [8] G. Brunner, Applications of Supercritical Fluids, in: J.M.D.M.F.S.M.A. Prausnitz (Ed.) *Annual Review of Chemical and Biomolecular Engineering*, Vol 1, 2010, pp. 321-342.
- [9] F. Sahena, I.S.M. Zaidul, S. Jinap, A.A. Karim, K.A. Abbas, N.A.N. Norulaini, A.K.M. Omar, Application of supercritical CO₂ in lipid extraction - A review, *Journal of Food Engineering*, 95 (2009) 240-253.
- [10] M.V. Palmer, S.S.T. Ting, Applications for supercritical fluid technology in food processing, *Food Chemistry*, 52 (1995) 345-352.
- [11] P.K. Rout, S. Naik, Y.R. Rao, Liquid CO₂ Extraction of Flowers of *Pandanus Fascicularis* Lam and Fractionation of Floral Concrete and Comparative Composition of the Extracts, *J. Food Biochem.*, 35 (2011) 500-512.
- [12] O.J. Catchpole, S.J. Tallon, W.E. Eltringham, J.B. Grey, K.A. Fenton, E.M. Vagi, M.V. Vyssotski, A.N. MacKenzie, J. Ryan, Y. Zhu, The extraction and fractionation of specialty lipids using near critical fluids, *J. Supercrit. Fluids*, 47 (2009) 591-597.
- [13] G. Brunner, Supercritical fluids: technology and application to food processing, *Journal of Food Engineering*, 67 (2005) 21-33.
- [14] J.A. Min, S.F. Li, J. Hao, N.H. Liu, Supercritical CO₂ Extraction of *Jatropha* Oil and Solubility Correlation, *J. Chem. Eng. Data*, 55 (2010) 3755-3758.
- [15] R. Halim, M.K. Danquah, P.A. Webley, Extraction of oil from microalgae for biodiesel production: A review, *Biotechnol. Adv.*, 30 (2012) 709-732.
- [16] F. Temelli, Perspectives on supercritical fluid processing of fats and oils, *J. Supercrit. Fluids*, 47 (2009) 583-590.
- [17] P.K. Rout, S. Naik, Y.R. Rao, Liquid CO₂ extraction of flowers and fractionation of floral concrete of *Michelia champaca* Linn, *J. Supercrit. Fluids*, 56 (2011) 249-252.
- [18] NIST Technology, Chemistry WebBook - Thermophysical Properties of Fluid Systems, 2011. Available at: <http://webbook.nist.gov/chemistry/>
- [19] J. Chrastil, Solubility of Solids and Liquids in Supercritical Gases, *J. Phys. Chem.*, 86 (1982) 3016-3021.
- [20] O. Doker, U. Salgin, N. Yildiz, M. Aydogmus, A. Calimli, Extraction of sesame seed oil using supercritical CO₂ and mathematical modeling, *Journal of Food Engineering*, 97 (2010) 360-366.
- [21] E. Reverchon, C. Marrone, Modeling and simulation of the supercritical CO₂ extraction of vegetable oils, *J. Supercrit. Fluids*, 19 (2001) 161-175.
- [22] Y.Y. Sun, S.F. Li, Measurement and correlation of the solubility of *Ligusticum Chuanxiong* oil in supercritical CO₂, *Chinese Journal of Chemical Engineering*, 13 (2005) 796-799.