Supercritical Fluid Extraction of Lipids from Deep-Fried Food Products

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ABSTRACT: A supercritical fluid extraction (SFE) method is described for extracting lipids from fried-food samples. Response surface analysis was used to study the effects of variables, including pressure, temperature, flow rate, and modifier (methanol) on lipid extraction by SFE. The analysis of variance for the response variables indicated that the models developed were satisfactory with coefficients of determination of 0.95 and 0.92 for chicken nuggets and potato fries, respectively. The models predicted that increasing the pressure increased the percentage lipid extracted for both chicken nuggets and potato fries. In addition, the pressure by temperature interactions were significant for chicken nuggets and potato fries. Slight differences in fatty acid composition were observed between SFE and the Goldfisch method. The SF extracts contained traces of $C_{12:0}$, $C_{20:0}$, and $C_{24:0}$ in chicken nuggets and $C_{14:1}$, $C_{18:3}$, $C_{22:0}$, and $C_{23:0}$ in potato fries, respectively, which are not found in the Goldfisch extracts. The optimal conditions for extraction are: 53 MPa, 150°C, 4 mL/min, and 10% modifier for chicken nuggets and 53 MPa, 150°C, 3 mL/min, and 0% modifier for potato fries. To duplicate the results of exhaustive Goldfisch extraction with petroleum ether, SFE conditions of 44 MPa, 80°C, 3 mL/min, and 0% modifier were used to produce similar results for both chicken nuggets and potato fries.
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KEY WORDS: Chicken nuggets, deep-fat frying, fatty acids, Goldfisch, lipid extraction, potato fries, RMS, response surface methodology, SFE, supercritical fluid extraction.

The United States Department of Agriculture and the Food and Drug Administration have recently revised the requirements for accurate labeling of fat in foods (1). In addition, the food industry requires a consistent determination of fat for quality-control purposes. Food analysts currently use techniques such as Soxhlet extraction with petroleum ether as the solvent (2) or methods involving acid hydrolysis followed by solvent extraction and nonheating methods (3).

Food manufacturers have looked for alternative methods of extraction, primarily to eliminate the use of toxic and flammable solvents and to reduce extraction time. Extraction of fats (i.e., triglyceride mixtures) can be readily accomplished with supercritical fluid extraction (SFE) by using supercritical carbon dioxide (SC-CO_2) as a solvent. This technique has been used in the extraction of fats and lipids from various matrices as demonstrated by a number of researchers (4–6). King and coworkers (7) used SFE on dehydrated foods and meats to extract free lipid fractions and total lipid. Lembke and Engelhardt (5) used SFE as an alternative to solvent extraction for total lipid determinations in meat sample matrices. This process has also been used to remove lipids and cholesterol from fish muscle (8). Results obtained on meats with fat content from 2–35% by weight indicate that over 96% of the fat content can be extracted by SC-CO_2 (8).

Nonpolar organic solvents, such as hexane and SC-CO_2, are suitable for the neutral or simple lipids, which include esters of fatty acids, acylglycerols, and unsaponifiable matter. Solid-phase extraction is particularly useful for complex polar lipids (9). However, polar lipids are not accessible to SC-CO_2. Although phospholipids are sparingly soluble in SC-CO_2, they can be recovered with ethanol as an entrainer (10).

Other factors that affect the selection of an acceptable solvent or an analytical method are food porosity, particle size of the food matrix, product moisture content, the extraction time, and addition of a modifier, such as ethanol or methanol (10–14). The time required to perform these extractions also depends on the sample lipid content and weight. However, sufficient information is not available in the literature to demonstrate the effectiveness of SFE to quantify lipid content from sources such as inherent fat in raw meat or absorbed fat in fried meats and nonfatty vegetable products.

The amount of lipid extracted depends on the solubility of triglycerides under given operating conditions. To characterize the extraction procedures, gas chromatography can be used to analyze the fatty acid profile of the extracted lipids. Studies carried out by Merkle and Larick (15) indicate that extraction conditions affected the fatty acid content of extracts.

The overall objective of this study was to examine and optimize the various parameters that affect lipid extraction from fried foods by SFE. Our study concentrated on fried chicken nuggets and potato fries. Raw chicken meat has an inherent fat content of 3 to 4% (16), with a significant amount of polar phospholipids (27 to 57% of total fat), and potatoes are low (0.1%) in fat (17).

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The specific objectives were: (i) to identify important operating conditions, develop predictive models, and determine optimal conditions for SFE of lipids from fried foods by response surface methodology (RSM); and (ii) to compare lipid extractions by SFE (under different conditions) and conventional Goldfisch methods.

EXPERIMENTAL PROCEDURES

Sample preparation. Two types of samples (chicken nuggets and potato fries), fried under commercial conditions, were purchased from local restaurants. The moisture from these samples was removed by drying in a vacuum-air oven at 70°C for 24 h by AACC method #44-40 (3). The samples were homogenized in a household blender prior to lipid extraction.

SFE procedure for lipid extraction. Lipid was extracted from fried foods in an SFE instrument (Model: Fatmaster; Suprex Corporation, Pittsburgh, PA), with a varipump SFE CO_2 pump and a variflow automatically variable restrictor. In the present study, the restrictor was preset to 75°C. Our preliminary experiments showed that a 20-min extraction time was sufficient to extract the lipid. Most of the lipid was extracted from the samples in the first 10 min, with the remaining lipid extracted in the next 10 min.

The dried sample was weighed into a 5-mL extraction vessel. Modifier (methanol), if necessary, was added as a percentage of the cell volume. The modifier was added to the sample in five steps to provide better migration of the modifier into the sample. The vessel was sealed and placed into the extraction oven for extraction of lipid. Lipid extractions from the samples were carried out two at a time in two separate vessels in a sequential mode. Following completion of the run, the vessels were removed, cooled, and weighed to determine the percentage of lipid in the sample.

Extraction of lipid by Goldfisch method. Moisture-free samples (2–3 g) were weighed into extraction thimbles and covered with glass wool. The thimbles were placed in glass sleeves and clamped into the lipid extractor (Model: 3500; Goldfisch, Labconco Corporation, Kansas City, MO). To the oil extraction beaker, 50 mL petroleum ether was added, and it was attached to the lipid extractor. The extraction was carried out for 16–24 h. After extraction, the beakers were placed in a vacuum oven at 70°C for 1 h to evaporate the petroleum ether and were then weighed, and the percentage lipid was calculated from the mass of lipid collected. Triplicate lipid analysis was performed for both samples.

Response surface analysis of SFE. The effect of operational pressure (P), oven temperature (T), flow rate (F), and modifier (M) concentration on the amount of lipid extracted were studied from a response surface analysis. A common experimental design, the Box-Behnken type (18), for investigating linear and quadratic effects of two or more factors was selected. The underlying principle of RSM is to find a simple mathematical expression (such as first- or second-order polynomials) to approximate as closely as possible the true relationship between response and factors (19). The conditions and their levels tested are given in Table 1. The complete design consisted of 27 experimental runs, including three replications (Experiment # 9, 18, and 27) at the center point. We observed that 150°C caused cooking of chicken nuggets during the extraction operation, so for potato fries the maximum operating temperature was reduced to 140°C.

The model was analyzed to fit the following second-order equation to all dependent variables:

$$Y = C_0 + C_1 \cdot P + C_2 \cdot T + C_3 \cdot F + C_4 \cdot M + C_5 \cdot PT + C_6 \cdot PF + C_7 \cdot PM + C_8 \cdot TF + C_9 \cdot TM + C_{10} \cdot MF + C_{11} \cdot P^2 + C_{12} \cdot T^2 + C_{13} \cdot F^2 + C_{14} \cdot M^2 \quad [1]$$

where Y is the response function (Y variables were assumed to be affected by the four independent variables), C_i's are constant regression coefficients and P, T, F, and M are independent variables. A second-order response surface prediction was fitted for each characteristic by using the statistical analysis system's RSREG procedure (20) and tested for adequacy and fitness by analysis of variance. Adequacy of the models at selected conditions was tested by performing independent experiments. The conditions were selected to include mini-

TABLE 1
The Experimental Design and Levels of Operational Parameters for Supercritical Fluid Extraction

		Pressure	Temperature[a]	Flow rate	Modifier
A—Coded and actual levels of operational parameters					
Code	–1	38.0 MPa	80 (80)°C	3.0 mL/min	0%
	0	45.6 MPa	115 (110)°C	4.0 mL/min	5%
	1	53.2 MPa	150 (140)°C	5.0 mL/min	10%
B—Experimental design in terms of coded levels					
Exp. #	1	1	1	0	0
	2	–1	1	0	0
	3	1	–1	0	0
	4	–1	–1	0	0
	5	0	0	1	1
	6	0	0	–1	1
	7	0	0	1	–1
	8	0	0	–1	–1
	9	0	0	0	0
	10	1	0	0	1
	11	–1	0	0	1
	12	1	0	0	–1
	13	–1	0	0	–1
	14	0	1	1	0
	15	0	–1	1	0
	16	0	1	–1	0
	17	0	–1	–1	0
	18	0	0	0	0
	19	1	0	1	0
	20	–1	0	1	0
	21	1	0	–1	0
	22	–1	0	–1	0
	23	0	1	0	1
	24	0	–1	0	1
	25	0	1	0	–1
	26	0	–1	0	–1
	27	0	0	0	0

[a]The values for temperature in parentheses were used for potato fries.

TABLE 2
Lipid Extraction Results from 27 Factorial Experiment for Chicken Nuggets and Potato Fries by Supercritical Fluid Extraction

Experiment number	Independent variable level[a]					Chicken nuggets[b]	Potato fries[b]
	P	*T1*	*T2*	*F*	*M*		
1	53.2	150	140	4.0	5	35.31	27.82
2	38.0	150	140	4.0	5	20.95	14.36
3	53.2	80	80	4.0	5	33.43	24.89
4	38.0	80	80	4.0	5	27.58	23.91
5	45.6	115	110	5.0	10	33.17	26.05
6	45.6	115	110	3.0	10	32.62	25.58
7	45.6	115	110	5.0	0	34.35	26.81
8	45.6	115	110	3.0	0	34.41	26.12
9	45.6	115	110	4.0	5	32.96	26.22
10	53.2	115	110	4.0	10	35.12	26.56
11	38.0	115	110	4.0	10	25.38	18.65
12	53.2	115	110	4.0	0	34.24	26.79
13	38.0	115	110	4.0	0	27.11	16.41
14	45.6	150	140	5.0	5	35.21	26.91
15	45.6	80	80	5.0	5	33.15	25.85
16	45.6	150	140	3.0	5	32.74	27.28
17	45.6	80	80	3.0	5	31.07	25.62
18	45.6	115	110	4.0	5	33.58	25.51
19	53.2	115	110	5.0	5	33.75	27.44
20	38.0	115	110	5.0	5	23.93	19.57
21	53.2	115	110	3.0	5	33.86	26.79
22	38.0	115	110	3.0	5	22.93	13.49
23	45.6	150	140	4.0	10	34.75	28.05
24	45.6	80	80	4.0	10	32.95	25.15
25	45.6	150	140	4.0	0	34.21	27.71
26	45.6	80	80	4.0	0	33.47	25.67
27	45.6	115	110	4.0	5	34.15	27.20

[a]Abbreviations: *P*, pressure (MPa); *T1*, temperature used for testing chicken nuggets (°C); *T2*, temperature used for testing potato fries (°C); *F*, flow rate (mL/min); *M*, modifier (%).
[b]Gram of fat extracted per 100 g of sample.

mum and maximum percentage lipid extraction. In addition, extraction conditions were selected so that the amount of lipid extracted was similar to that from Goldfisch. The deviation between the predicted and experimental values was calculated from the root mean square error (RMS):

$$RMS = \sqrt{\frac{\sum (Y_P - Y_O)^2}{n}} \qquad [2]$$

where Y_P is the value of predicted lipid extracted from the model, Y_O is the value of lipid extracted experimentally for the corresponding extraction conditions, and n is the number of extraction conditions.

Fatty acid analysis. The lipid samples, extracted by SFE and Goldfisch, were first transesterified to fatty acid methyl esters (FAME) with boron trifluoride in methanol as reported by Morrison and Smith (21) and then measured by capillary gas chromatography (GC) in a Model 5790 gas chromatograph (Hewlett-Packard, Avondale, PA). A flame-ionization detector was used. Samples were analyzed on a capillary column (Omegawax™ 250, 30 m × 0.25 µm i.d. × 0.25 µm film; Supelco Co., Bellfonte, PA) to identify key fatty acids with Supelco ™ 37 FAME mix as standard. This standard mix contains FAME that range in carbon number from C_4 to $C_{24:1}$, including most of the important saturated, monounsaturated, and polyunsaturated FAME. The GC was programmed from 180°C (2 min) to 220°C at 4°C/min, with a hold of 23 min, and helium was used as the carrier gas (30 cm/s, 205°C). The samples (3 µL, split mode 100:1) were injected onto the column.

RESULTS AND DISCUSSION

Lipid contents of food samples, determined by the Goldfisch method, were 32.98 ± 0.26 and 26.0 ± 0.31% for chicken

TABLE 3
Analysis of Variance

Source	DF[a]	Sum of squares[b]	
		Chicken nuggets	Potato fries
Model	14	424.28*	412.80*
Linear	4	283.01*	247.31*
Quadratic	4	120.79*	117.42*
Crossproduct	6	20.48	48.07
Residual	12	20.06	34.24
Lack of fit	10	17.52	32.80
Pure error	2	2.54	1.44
R^2		0.95	0.92

[a]Degrees of freedom.
[b]Values significant at α = 0.05 are identified with an asterisk (*).

nuggets and potato fries, respectively. The SFE results (Table 2) were similar to the Goldfisch data for both products and were within ±10% of Goldfisch values, except at 38 MPa (code level = −1) where the percentage lipid extracted was lower.

Equation 1 was fitted to the experimental data (Table 2) and was tested for adequacy of fitness by analysis of variance. The results are summarized in Table 3. The models developed were appropriate and adequate with satisfactory coefficient of determination (R^2) value (chicken nuggets, 0.95; potato fries, 0.92) and possessed no significant lack of fit. The analysis of variance for response (percentage lipid extraction) indicates that the model was significant ($P < 0.05$) for chicken nuggets and potato fries.

The estimated regression coefficients and the results of significance tests on the coefficients are indicated in Table 4. The significance tests on the estimates showed that, in both chicken nuggets and potato fries, pressure and temperature were the two important process variables with significant effects. For potato fries, the pressure by temperature interaction was significant for lipid extraction, with flow and modifier having little or no effect. Previous studies by Spanos *et al.* (22) also indicated that higher yields were obtained with higher pressures. Experiments conducted by King *et al.* (7) at 35–70 MPa and 80°C have shown that dense CO_2 was an effective agent for selective removal of fat from a variety of meat matrices.

TABLE 4
Least Square Estimates[a] of Model Parameters and Standard Errors for Chicken Nuggets and Potato Fries

Parameter	Chicken nuggets	Potato fries
Intercept	−123.3 ± 35.35*	−120.3 ± 48.12*
Pressure (*P*)	6.46 ± 0.99*	6.41 ± 1.31*
Temperature (*T*)	−0.32 ± 0.17	−0.71 ± 0.27*
Flow rate (*F*)	7.98 ± 6.34	11.62 ± 8.39
Modifier (*M*)	−1.36 ± 1.05	0.65 ± 1.4
$P{\cdot}P$ ($\times 10^{-4}$)	−0.073 ± 0.0097*	−0.072 ± 0.013*
$T{\cdot}P$ ($\times 10^{-4}$)	0.008 ± 0.0024*	0.014 ± 0.0037*
$T{\cdot}T$ ($\times 10^{-4}$)	−0.0003 ± 0.0005	0.0005 ± 0.0008
$F{\cdot}P$	−0.036 ± 0.085	−0.179 ± 0.111
$F{\cdot}T$ ($\times 10^{-4}$)	0.003 ± 0.018	−0.005 ± 0.028
$F{\cdot}F$	−0.79 ± 0.56	−0.278 ± 0.73
$M{\cdot}P$ ($\times 10^{-4}$)	0.017 ± 0.017	−0.016 ± 0.022
$M{\cdot}T$ ($\times 10^{-4}$)	0.0015 ± 0.0037	0.001 ± 0.006
$M{\cdot}F$	0.031 ± 0.13	−0.011 ± 0.169
$M{\cdot}M$	0.022 ± 0.022	−0.0009 ± 0.029

[a]Values significant at $\alpha = 0.05$ are identified with an asterisk (*).

Several researchers have reported that modifier concentration in CO_2 is important in contributing to the increase in extraction. Temelli (10) mixed the sample (canola meal) with ethanol and left it overnight at 4°C to equilibrate before SC-

TABLE 5
Fatty Acid Composition of Lipid Extracted from Chicken Nuggets by Supercritical Fluid Extraction (SFE) and Goldfisch (GF)

	SFE						GF
Conditions[a]	1	2	3	4	5	6	
P	44.6	38.0	53.2	43.6	45.1	44.1	
T	80	80	150	150	80	115	
F	4.0	3.0	3.0	4.0	4.0	4.0	
M	0	0	0	0	5	5	
Lipid extracted (%)							
Predicted	34.21	26.97	35.39	32.31	33.24	32.59	n/a
Observed	32.88	22.24	36.04	27.99	32.37	33.38	32.98
Fatty acid profile (relative %)							
$C_{12:0}$	0.03	0.41	0.03				
$C_{14:0}$	0.48	0.42	0.37	0.38	0.78	0.63	0.44
$C_{14:1}$	0.10		0.04				0.07
$C_{15:0}$	0.05	0.08	0.05	0.04			0.05
$C_{16:0}$	20.84	20.18	18.1	18.24	28.76	21.26	19.44
$C_{16:1}$	3.69	2.24	2.27	2.10	3.30	4.18	3.03
$C_{17:0}$	0.13	0.12	0.12	0.11			0.14
$C_{17:1}$	0.08		0.04				0.06
$C_{18:0}$	5.63	5.63	5.63	5.63	5.63	5.63	5.63
$C_{18:1}$	35.79	23.22	26.19	30.67	31.92	36.31	34.29
$C_{18:2}$	28.25	14.09	16.76	14.21	17.11	29.09	23.12
$C_{18:3n\text{-}6}$	0.20	0.07	0.08				0.15
$C_{18:3n\text{-}3}$	1.55	0.59	0.78	0.63	0.54	1.29	1.08
$C_{20:0}$	0.27	0.21	0.23	0.21			
$C_{20:1}$	1.99	3.42	2.16	3.14	2.38		1.4
$C_{22:0}$	0.27	0.19	0.20	0.19			0.25
$C_{24:0}$		0.50	0.36				

[a]Abbreviations: *P*, pressure (MPa); *T*, temperature (°C); *F*, flow rate (mL/min); *M*, modifier; n/a, not applicable.

TABLE 6
Fatty Acid Composition of Lipid Extracted from Potato Fries by Supercritical Fluid Extraction (SFE) and Goldfisch (GF)

	SFE			GF
Conditions[a]	1	2	3	
P	43.6	53.2	46.6	
T	110	140	80	
F	4.0	5.0	5.0	
M	5	0	10	
Lipid extracted (%)				
Predicted	24.82	29.57	26.50	n/a
Observed	26.51	28.38	26.54	25.31
Fatty acid (relative %)				
$C_{14:0}$	0.22	0.20	0.28	0.28
$C_{14:1}$		0.52		
$C_{16:0}$	17.15	15.22	19.07	17.21
$C_{17:0}$	0.13	0.17		0.20
$C_{18:0}$	12.80	12.80	12.80	12.80
$C_{18:1}$	45.13	48.41	41.11	41.41
$C_{18:2}$	4.39	5.12	3.50	3.68
$C_{18:3n-6}$		0.29		
$C_{18:3n-3}$		0.19		
$C_{20:0}$	0.22	0.29		0.24
$C_{22:0}$		0.24		
$C_{23:0}$		0.93		

[a]*P*, pressure (MPa); *T*, temperature (°C); *F*, flow rate (mL/min); *M*, modifier.

CO_2 extraction. A mixture of CO_2 and ethanol was pumped through the sample (carrot press cake) in studies conducted by Vega *et al*. (23). Levy and coworkers (24) studied the effect of ethanol on extraction efficiency, and ethanol was added to the samples (animal feeds and snack foods) with a modifier addition pump. These studies indicate a positive effect of the modifier on extraction efficiency. However, in our experiments, the modifier effect was not significantly correlated for lipid extraction from either chicken nuggets or potato fries. This may be due to the way in which the modifier was added to the sample, or the type of modifier (methanol) used.

Contour plots. Contour plots of percentage lipid extracted were generated by using the model to illustrate the predicted responses with varying levels of pressure and temperature, while flow rate and modifier were held at a constant level. Contour plot representation of the percentage lipid extracted for chicken nuggets and potato fries as a function of pressure and temperature, developed for constant flow rate values of 3, 4, and 5 mL/min and modifier concentrations of 0, 5, and 10%, respectively, is shown in Figures 1 and 2. On the contour plot surface, the predicted values increase gradually from left to right, and numbers on the contour lines indicate the fat extraction values. The contour lines for three temperatures show that an increase in pressure results in higher extraction values.

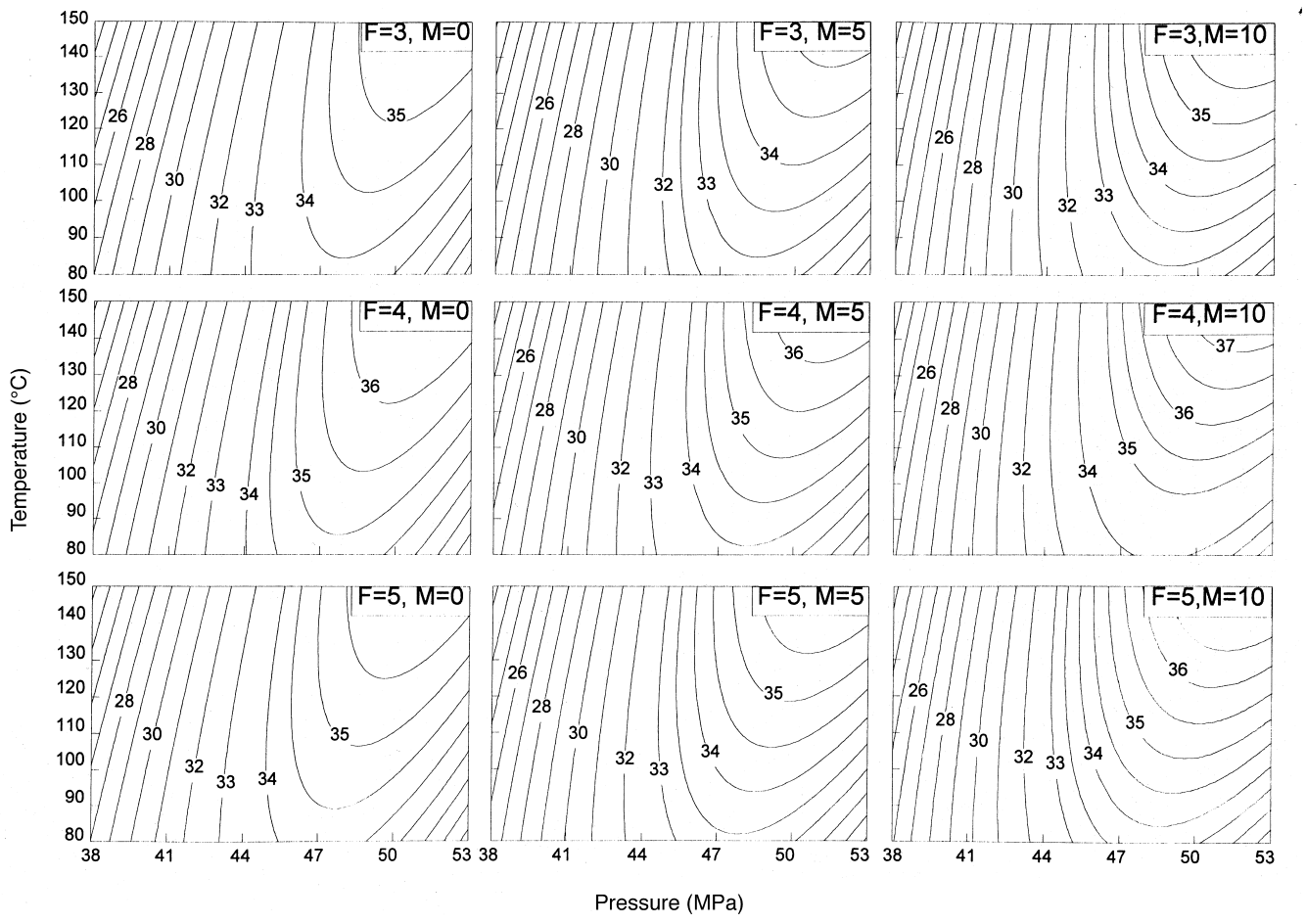

FIG. 1. Response surface plots of percentage fat extracted from chicken nuggets for various flow rates (*F*, mL/min) and modifier concentrations (*M*, %).

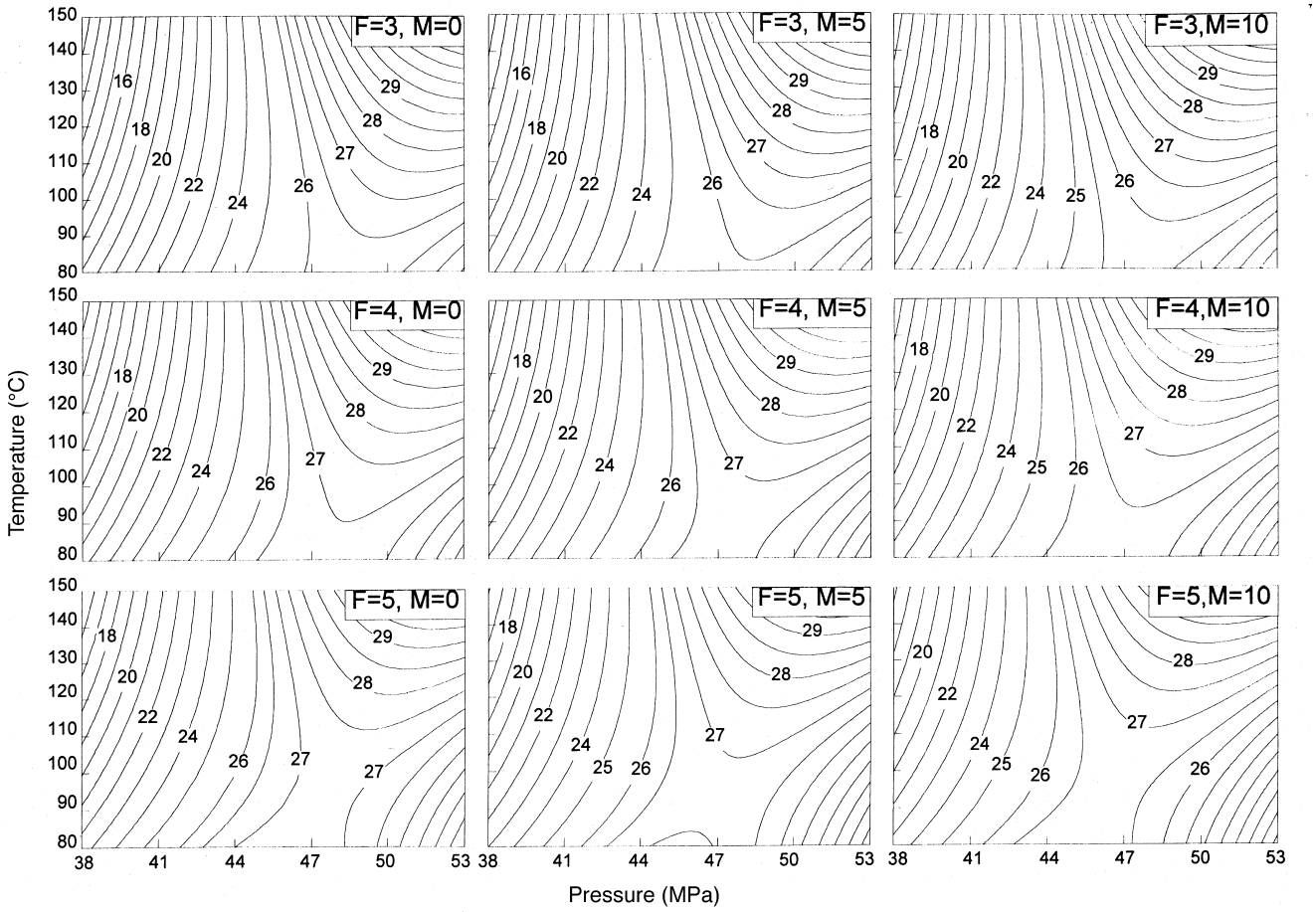

FIG. 2. Response surface plots of percentage fat extracted from potato fries for various flow rates (*F,* mL/min) and modifier concentrations (*M,* %).

From the shape of the surface (Fig. 1), it is apparent that higher yields are possible with pressure and temperature regardless of the presence of modifier. Increasing pressure increases SC-CO_2 density, which enhances extraction efficiency. At any given pressure and temperature, modifier and flow rate showed no significant effect.

From the model predictions (Figs. 1 and 2) it is apparent that increasing the flow rate extracts the same amount of lipid at a lower pressure. Addition of a modifier showed a slight negative effect at all levels of flow rate for potato fries and a slight increase for chicken nuggets.

In both chicken nuggets and potato fries, increasing the temperature at low pressure decreased the amount of lipid extracted (Figs. 1 and 2). However, increasing the temperature at high pressure increased the amount of lipid extracted. This may be due to the increased solubility of lipid in SC-CO_2. This marked difference can be attributed to the enhanced solubility of the component triglycerides in CO_2 at higher gas pressures (25,26).

Predicted response. Adequacy of the models at selected conditions was tested by performing independent experiments (Tables 5 and 6). Most of the observed extraction values for chicken nuggets as well as potato fries agreed well with their predicted values. The predictions (RSM values) were within ±2.73% for chicken nuggets and ±1.19% for potato fries.

GC. Fatty acid profiles of lipids from these selected conditions were obtained to observe the differences among extraction conditions. Fatty acids are reported as relative percentages of the entire fatty acid profile (Table 5). Unidentified peaks were not considered because they may be degradation products of the frying oil. The data were normalized to 100% by including only the identified peaks. In chicken nuggets, $C_{16:0}$, $C_{18:1}$, and $C_{18:2}$ were the major fatty acids in the Goldfisch as well as the SC-CO_2 extracts. Chicken nuggets contained traces of $C_{12:0}$, $C_{20:0}$, and $C_{24:0}$.

Longer-chain fatty acids, such as $C_{20:0}$, $C_{22:0}$, and $C_{24:0}$, were not present in the SC-CO_2 lipid extracts when methanol was used. There was a slight increase in the extraction of longer-chain fatty acids, such as $C_{20:0}$ and $C_{24:0}$, under certain conditions in SFE as compared to Goldfisch.

The fatty acid composition of the lipid extracts from potato fries by the Goldfisch method was identified (Table 6). Carbon chainlength varied between $C_{14:0}$ to $C_{23:0}$ for the fatty acids found in potato fries. The major fatty acids found in both Goldfisch and SFE extracts were $C_{16:0}$, $C_{18:0}$, and $C_{18:1}$. Differences were noticed in the fatty acid profile of the potato

fries that were extracted under higher operational conditions (pressure and temperature). This lipid extract exclusively contained traces of $C_{14:1}$, $C_{18:3}$, $C_{22:0}$, and $C_{23:0}$. These fatty acids were not present in the Goldfisch extracts. This change in composition may be due to the differences in solubility of fatty acids in SC-CO_2.

Under optimized conditions, quantitative lipid extraction could be performed in about 30 min (compared to 8–16 h by the conventional method). Overall, the similarity between observed and predicted values indicated that the statistical model was useful and that it could be used for different fried foods. Results from RSM show that pressure and a combination of pressure and temperature contribute significantly to the extraction efficiency of chicken nuggets and potato fries by the SFE method.

These results suggest that extraction with SC-CO_2 is a viable technique for reducing the generation of large quantities of solvents and their disposal problem. It appears that the best conditions for extraction are 53 MPa, 150°C, 4 mL/min, and 10% modifier for chicken nuggets. Similarly, for potato fries, the conditions are 53 MPa, 150°C, 3 mL/min, and 0% modifier. Compared to conventional lipid extraction procedures, SFE conditions of 44 MPa, 80°C, 3 mL/min, and 0% modifier produced similar results for both products.

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REFERENCES

1. Kurtzweil, P., Nutrition Facts—Focus on Food Labeling, *FDA Consumer 27*:22–27 (1993).
2. AOAC, *Official Methods of Analysis,* 15th edn., AOAC International, Arlington, 1990.
3. AACC, *Approved Methods of the American Association of Cereal Chemists*, 8th edn., American Association of Cereal Chemists, St. Paul, 1983.
4. Fattori, M., N.R. Bulley, and A. Meisen, Fatty Acid and Phosphorus Contents of Canola Seed Extracts Obtained with Supercritical Carbon Dioxide, *J. Agric. Food Chem. 35*:739–743 (1987).
5. Lembke, P., and H. Engelhardt, Development of a New SFE Method for Rapid Determination of Total Fat Content of Food, *Chromatographia 35*:509–516 (1993).
6. Hampson, J.W., K.C. Jones, T.A. Foglia, and K.M. Kohout, Supercritical Fluid Extraction of Meat Lipids: An Alternative Approach to the Identification of Irradiated Meats, *J. Am. Oil Chem. Soc. 73*:717–721 (1996).
7. King, J.W., J.H. Johnson, and J.P. Friedrich, Extraction of Fat Tissue from Meat Products with Supercritical Carbon Dioxide, *J. Agric. Food Chem. 37*:951–954 (1989).
8. Hardardottir, K., and J.E. Kinsella, Extraction of Lipid and Cholesterol from Fish Muscle with Supercritical Fluids, *J. Food Sci. 53*:1656–1658,1661 (1988).
9. Mossoba, M.M., and David Firestone, New Methods for Fat Analysis in Foods, *Food Testing Anal. 2*:24–32 (1996).
10. Temelli, F., Extraction of Triglycerides and Phospholipids from Canola with Supercritical Carbon Dioxide and Ethanol, *J. Food Sci. 57*:440–442,457 (1992).
11. Bhatty, R.S., Comparison of the Soxtec and Goldfisch Systems for Determination of Oil in Grain Species, *Can. Inst. Food Sci. Technol. J. 18*:181–184 (1985).
12. McGhee, J.E., L.T. Black, and O.L. Brekke, Fat Content in Corn Grits: Effects of Grinding, Extraction Solvents, and Analytical Methods, *Cereal Chem. 51*:472–477 (1974).
13. Hubbard, W.D., A.J. Sheppard, D.R. Newkirk, A.R. Prosser, and T. Osgood, Comparison of Various Methods for the Extraction of Total Lipids, Fatty Acids, Cholesterol and Other Sterols from Food Products, *J. Am. Oil Chem. Soc. 54*:81–83 (1977).
14. Ngeh-Ngwainbi, J., and R.H. Lane, Perspectives on Fat Content, *Cereal Foods World 39*:82–91 (1994).
15. Merkle, J.A., and D.K. Larick, Fatty Acid Content of Supercritical Carbon Dioxide Extracted Fractions of Beef Fat, *J. Food Sci. 60*:959–962 (1995).
16. Pikul, J., and F.A. Kummerow, Lipid Oxidation in Chicken Muscles and Skin After Roasting and Refrigerated Storage of Main Broiler Parts, *Ibid. 55*:30–37 (1990).
17. Stauffer, C.E., Frying Fats, in *Fats and Oils*, The American Association of Cereal Chemists, Inc., St. Paul, 1996, pp. 83–84.
18. Box, G.E.P., and D.W. Behnken, Some New Three Level Designs for the Study of Quantitative Variables, *Technometrics 2*:455–476 (1960).
19. Mead, R., and D.J. Pike, A Review of Response Surface Methodology from a Biometric Viewpoint, *Biometrics 31*:803–851 (1975).
20. SAS, *SAS User's Guide: Statistics*, Version 5 Edition, SAS Institute Inc., Cary, 1985.
21. Morrison, W.R., and L.M. Smith, Preparation of Fatty Acid Methyl Esters and Dimethylacetals from Lipids with Boron Fluoride–Methanol, *J. Lipid Res. 5*:600–608 (1964).
22. Spanos, G.A., H. Chen, and S.J. Schwartz, Supercritical CO_2 Extraction of β-Carotene from Sweet Potatoes, *J. Food Sci. 58*:817–820 (1993).
23. Vega, P.J., M.O. Balaban, C.A. Sims, S.F. O'Keefe, and J.A. Cornell, Supercritical Carbon Dioxide Extraction Efficiency for Carotenes from Carrots by RSM, *Ibid. 61*:757–759, 765 (1996).
24. Levy, J.M., V. Danielson, R. Ravey, and L. Dolata, Routine Determination of Fats in Foods Using Supercritical Fluid Extraction, *Liquid Chrom. Gas Chrom. 12*:920–923 (1994).
25. Friedrich, J.P., G.R. List, and A.J. Heakin, Petroleum-Free Extraction of Oil from Soybeans with Supercritical CO_2, *J. Am. Oil Chem. Soc. 59*:288–292 (1982).
26. Stahl, E., E. Schütz, and H.K. Mangold, Extraction of Seed Oils with Liquid and Supercritical Carbon Dioxide, *J. Agric. Food Chem. 28*:1153–1157 (1980).

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