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Optimization of Extraction of Wax from Flax Straw by Supercritical Carbon Dioxide

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Response surface methodology (RSM) was used to optimize supercritical carbon dioxide (SC-CO_2) extraction of wax from flax straw. SC-CO_2 variables included flow rate (24.8, 35, 50, 65, and 75.2 g/min), pressure (132, 200, 300, 400, and 468 bar), and temperature (34.8, 45, 60, 75, and 85.2°C). The SC-CO_2 extraction technique was optimized for the extraction of flax straw wax and its main constituents (fatty acids, alkanes, sterols, aldehydes, and wax esters). A second order polynomial model provided a good fit (R^2 value of 0.90) for the yield of wax from flax straw. Canonical analysis revealed a maximum stationary point for wax yield of 1.26 g/100 g at a flow rate of 61.1 g/min, a pressure of 378 bar and a temperature of 75°C. The fatty acid and wax ester contents of flax wax were also fitted with a second order polynomial model with R^2 values of 0.94 and 0.87, respectively. The temperature was found to be the most important factor affecting the recovery of wax. Linear and quadratic effects of temperature and interaction effects of temperature and pressure had a significant effect on the total wax yield and on the content of fatty acids, fatty alcohols, and sterols in the wax.

Keywords flax straw; optimization; response surface methodology; supercritical carbon dioxide; wax

INTRODUCTION

Natural waxes obtained from sugar cane (1), orange peel (2), rice bran (3), and sorghum grain (4) are of interest due to their applications in the cosmetic and pharmaceutical industries. When considering a new raw material, cosmetic producers take into account the active chemical functionality, quality, production technology, and cost implications of the material (2) and increasingly by-products of industrial processes and agricultural residues are receiving more attention as potential sources of natural wax. Straw (flax and wheat) is mainly composed of cellulose, hemicellulose, lignin, and silica (5). Also, phenolic compounds, like ferulic coumaric, syringic, vanillic acid, and vanillin have been reported to be present in herbaceous straw (5).

Valuable wax compounds from wheat (0.56% of yield at 300 bar and 40°C) (6), flax (2.5% of yield from cv Flanders at 300 bar and 70°C) (7–8), and triticale straw (1.1% of yield at 350 bar and 40°C) (9) have been fractionated through the SC-CO_2 technique and are currently being investigated further. Waxes obtained from flax and wheat have shown suitable chemical and thermal properties (melting point, crystallization, decomposition, heat capacity, and oxidative stability) and are thus potential replacements for products currently used in cosmetics, foods, and pharmaceuticals. The major components of the SC-CO_2 extracts of flax waxes are fatty acids (36–49%), fatty alcohol (20–26%), aldehydes (10–14%), wax esters (5–12%), sterols (7–9%), and alkanes (4–5%) (7). Flax and wheat straw waxes are a good source of policosanol and sterols, both of which are known to have cholesterol-lowering properties. Therefore, extraction of wax from straw could add significant value to the straw and enhance the economic viability of conversion of straw into bioenergy and biochemicals.

Recently, SC-CO_2 extraction technology has been incorporated into a biorefinery potentially enabling straw to be converted into a variety of high value wax compounds using SC-CO_2 methodology (6–10). Dewaxed wheat straw produced by SC-CO_2 has shown promising results in the generation of electricity and production of strawboard, green mulch, and paper pulp (10). Thus, it may be possible to convert straw into energy and a wide range of industrial products using SC-CO_2 extraction technology.

Supercritical fluid extraction has many benefits over traditional extraction techniques (6–7). SC-CO_2 is a preferred solvent in food extractions because it is non-toxic, non-explosive, easily separated, and has a low critical temperature and price (10–11). Due to its low critical temperature (31.1°C), SC-CO_2 is ideal for the extraction of compounds which decompose at high temperature. The ease of fine-tuning operating conditions to increase extraction makes this technology a good option for the recovery of natural compounds (12). Crude palm oil (13), β-carotene (14), tocopherol (15), squalene (16), and sterols (17) are some of the compounds that have been successfully obtained using SC-CO_2 extraction.

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In a previous study, we successfully utilized SC-CO_2 extraction techniques to extract wax from flax straw (7). However, successful application of SC-CO_2 technology requires a fundamental understanding and optimization of the process variables. Pressure, temperature, and CO_2 flow rate are important variables in SC-CO_2 extraction (11–18). Response surface methodology (RSM) has been widely utilized in the food processing industry to optimize the yield of desired compounds and to understand the effect of process variables. RSM uses mathematics and statistics to generate models that describe the process, analyze effects of independent variables, and optimize process operations (19).

The primary objective of this investigation was to determine the optimal pressure, temperature, and flow rate conditions for SC-CO_2 extraction of wax from flax straw. The extraction of high value components of the extracted wax, including fatty acids, fatty alcohols, and sterols, was also optimized.

MATERIALS AND METHODS

Materials

CDC Bethune flax (*Linum usitatissimum L.*) straw was provided by ABIP-CBioN, Agriculture Agri-Food Canada, Swift Current, SK. The straw was milled (Wiley Mill, Arthur H. Thomas Co., Philadelphia, PA) to pass through a 2 mm screen and extracted under different conditions. Hexane, toluene, ethanol, *bis*-(trimethylsilyl)-trifluoroacetamide with 1% trimethylchlorosilane, alkane (C8 to C20 and C21 to C40), and fatty alcohol standards (C26 to C30) were purchased from Sigma–Aldrich Canada Ltd. (Oakville, ON). A mixture of plant sterols (brassicasterol, campesterol, stigmasterol, and β-sitosterol) and fatty acids (FIM-FAME-7 mix) were purchased from Matreya LLC (Pleasant Gap, PA).

Experimental Design

RSM with a central composite design (CCD) was conducted to investigate the effect of flow rate, pressure, and temperature of SC-CO_2 on the extraction of wax from flax straw. Variables and coded levels of CCD are shown in Table 1. Lower, middle, and upper levels of SC-CO_2 extraction were selected based on previous experiments (7–9), literature data (6), and the limitations of the SC-CO_2 apparatus. All experiments were conducted randomly to minimize the effects of unexpected variability due to extraneous factors. The CCD consisted of 18 experimental runs with four replicates at the center point (Table 1). Total wax yield and major wax components (fatty acids, alkanes, fatty alcohols, sterols, aldehydes, and wax esters) were used to develop a second order regression model capable of predicting optimal extraction conditions, including the flow rate, pressure, and temperature.

TABLE 1
Central composite experimental design for the extraction of wax from flax straw by SC-CO_2

		Factor 1 (X_1)	Factor 2 (X_2)	Factor 3 (X_3)
Run	Randomized run order	Flow rate (g/min)	Pressure (bar)	Temperature (°C)
1	5	35 (−1)[a]	200 (−1)	45 (−1)
2	15	35 (−1)	200 (−1)	75 (+1)
3	18	35 (−1)	400 (+1)	45 (−1)
4	12	35 (−1)	400 (+1)	75 (+1)
5	13	65 (+1)	200 (−1)	45 (−1)
6	16	65 (+1)	200 (−1)	75 (+1)
7	7	65 (+1)	400 (+1)	45 (−1)
8	17	65 (+1)	400 (+1)	75 (+1)
9	9	24.8 (−1.68)	300 (0)	60 (0)
10	11	75.2 (+1.68)	300 (0)	60 (0)
11	1	50 (0)	132 (−1.68)	60 (0)
12	4	50 (0)	468 (+1.68)	60 (0)
13	14	50 (0)	300 (0)	34.8 (−1.68)
14	10	50 (0)	300 (0)	85.2 (+1.68)
15	3	50 (0)	300 (0)	60 (0)
16	6	50 (0)	300 (0)	60 (0)
17	8	50 (0)	300 (0)	60 (0)
18	2	50 (0)	300 (0)	60 (0)

[a]Numbers in parentheses indicate coded values of independent variables in the experimental design.

The second order equation is:

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3$$

where Y is the predicted response; β_0 is the intercept; β_1, β_2, and β_3 are the linear coefficients; β_{11}, β_{22}, and β_{33} are the squared coefficients; β_{12}, β_{13}, and β_{23} are the interaction coefficients. Second order polynomial model, regression analysis, contour plots, and canonical analysis were studied using the Statistical Analysis System (SAS, version 8.1). Three-dimensional surface response plots were generated with SigmaPlot v 8.0 (SPSS Inc., Chicago, IL) using two variables within the experimental range and holding the other constant at a fixed point.

Supercritical CO_2 Extraction of Flax Straw

Extraction with SC-CO_2 was performed using a Thar Technology (Pittsburgh, PA) SFE-0.5 L system, equipped with a P-200A high pressure pump, a BPR-A200 backpressure regulator, an electric heater, a temperature controller, sample holders, and a computer for controlling the extraction temperature, time, and pressure (7). Liquid CO_2 from a cylinder (99.95% purity, Praxair, Edmonton, AB) was used as the solvent. After an initial air purge, a 100 g sample of flax straw was placed in the extraction vessel. Extraction temperature, pressure, and flow rate were controlled by computer software and maintained for 90 min. When the desired temperature, pressure, and CO_2 flow rate were achieved, extraction began. Lipid-laden solvent from the extractor was passed through a heated metering valve where SC-CO_2 was depressurized. Extracted wax was collected in a stainless steel vessel, and CO_2 was vented out of the system. The extract was flushed with nitrogen for 24 h to achieve a constant weight and stored in the dark at -40°C until analysis. Extract yield was determined by weighing the total extract (gravimetrically) using the equation:

$$\text{Total wax yield }(\%) = [\text{Dry extract (g)}/\text{Feed flax straw (g)}] \times 100.$$

GC/MS Analysis

The extracts (3 mg) were silylated with 200 µL of *bis*-(trimethylsilyl)-trifluoroacetamide with 1% trimethylchlorosilane (TMCS) in 100 µL of toluene at 75°C for 30 min. The reaction mixture was allowed to cool, evaporated under N_2, and re-dissolved in hexane for analysis. GC/MS analysis was conducted with an Agilent 6890/5973 GC-MS network gas system (Agilent Technologies, Wilmington, DE) equipped with a splitless injector, a network mass selective detector, and 7683 series Agilent auto-sampler. The high-temperature capillary column used for analysis was a DB-17HT (30 m x 0.25 mm I.D., 0.1 µm film thicknesses, J & W Scientific, Folsom, CA). Helium was used as a carrier gas (1.3 mL/min). The temperature of the injector was maintained at 300°C and the flow rate was 1.3 mL/min. An initial oven temperature of 50°C (1 min) was increased at a rate of 10°C/min to 280°C and then increased to 350°C at 5°C/min and maintained at this temperature for 10 min (7). An Agilent 5973 quadrupole mass spectrometer was operated in electron ionization (EI) mode at 70 eV, a source temperature of 280°C, a quadrupole temperature of 150°C, and a scan range of 35 to 350 m/z. Data were collected with Agilent enhanced ChemStation software (standard MSD version) and searched against the NIST (v. 02) and Wiley (v. 138) libraries (Palisade Corp., Newfield, NY). Compounds were identified by comparing obtained spectra with those in the library and with authentic standards. Octacosanol (fatty alcohol), stigmasterol (sterols), octacosane (alkane and aldehydes), myristic acid (fatty acid), and behenyl behenate (wax ester) were used as external standards to calculate the sample amount.

RESULTS AND DISCUSSION

Effect of SC-CO_2 Parameters on the Yield and Composition of Wax

The experimental design, including the range of independent variables and relevant coded values, are shown in Table 1. The range of independent variables and middle points were selected based on previous experimental results and data from the literature. The yield, major components of wax, and effects of variables (flow rate, pressure, and temperature) are shown in Table 2. The yield and composition of wax were considerably different after each extraction. The yield of wax ranged from 0.52 to 1.23%. These results are in agreement with our previous results on SC-CO_2 extraction (300 bar at 70°C for 90 min) of flax straw (NorLin, Flanders and AC McDuff), yielding 0.6 to 2.5% wax (7).

According to surface analysis results, the temperature and the pressure had a significant ($p < 0.01$) effect on the extraction of wax from flax straw. Temperature and pressure have a direct effect on the density of SC-CO_2; thus, complete extraction of the target compounds can be achieved by proper manipulation of both parameters. Also, in this study, the flow rate had a significant effect ($p < 0.05$) on the extraction of wax from flax straw.

The fatty acid content of flax waxes has been shown to vary between 118 and 256 mg/100 g (Table 2). Wax from three cultivars of flax (NorLin, Flanders and AC McDuff) extracted using SC-CO_2 at 300 bar, 70°C for 90 min, had fatty acids as the most abundant constituent (7). Fatty acids were significantly affected by the flow rate, temperature and pressure ($p < 0.01$). Morrison et al. (8) obtained 135 and 125 mg/g of fatty acids from flax dust with SC-CO_2 (552 bar and 60°C for 60 min) and hexane extraction, respectively. Additionally, Athukorala et al. (7) reported 374 to 639 mg/100 g of fatty acids from flax straw dried under different conditions.

The primary alcohol content of flax wax ranged from 48 to 99 mg/100 g (Table 2) and was composed of C26, C28, and C30 homologues (7). Sugarcane leaves are composed of 181 mg/Kg of fatty alcohols, while wheat straw contains 164 mg/Kg (20). SC-CO_2 extraction pressure had a significant effect ($p < 0.1$) on the yield of fatty alcohols. The alkane content of flax wax was found to be in the range of 19 to 75 mg/100 g (Table 2). Gutierrez and Rio (21) reported 35 to 84 mg/100 g of alkanes in flax fibers and their alkaline pulps. According to Athukorala et al. (7) and Gutierrez and Rio (21), the most abundant alkane in the wax of flax straw is nonacosane. Temperature had a significant ($p < 0.05$) effect on the extraction of total alkanes; however, the flow rate and pressure were insignificant.

Stigmasterol, β-sitosterol, and campesterol were the main sterols present in flax wax (7, 21), and the content of total sterols varied between 44 and 98 mg/100 g

TABLE 2
Experimental data for wax yield and components extracted under different $SC\text{-}CO_2$ process conditions from flax straw

Run	Total yield (g/100 g)	Main compounds (mg/100 g)					
		Fatty acids	Primary alcohol	Alkanes	Sterols	Aldehydes	Wax esters
1	0.75	121.92	70.06	32.24	63.68	44.48	6.46
2	0.74	129.70	61.12	28.09	68.68	27.21	10.65
3	0.71	136.13	77.31	27.27	68.42	7.97	16.34
4	0.91	209.43	95.36	40.78	76.85	55.66	23.24
5	0.75	192.99	78.93	36.26	76.97	59.86	10.42
6	0.92	193.28	89.53	36.15	66.17	12.58	46.48
7	0.84	177.21	78.09	31.07	69.46	39.49	17.10
8	1.21	246.89	85.92	54.60	97.59	74.75	38.43
9	0.84	138.18	90.54	40.38	60.61	71.15	17.87
10	0.96	222.13	63.94	27.95	80.11	24.43	28.17
11	0.52	118.55	48.91	19.08	52.09	31.97	10.25
12	1.10	216.45	95.40	38.59	75.18	40.43	27.67
13	0.62	127.61	64.20	26.26	43.68	3.84	12.27
14	1.12	253.13	63.14	74.78	75.52	20.18	21.97
15	1.12	231.13	99.09	38.15	89.53	24.87	26.73
16	1.07	241.63	97.45	38.28	89.06	22.67	26.32
17	1.23	255.71	97.75	39.24	82.07	22.72	25.64
18	1.17	226.51	98.06	44.27	89.24	25.79	30.28
Main effects							
Flow rate	**	***	NS	NS	NS	NS	**
Pressure	***	***	*	NS	*	**	*
Temperature	***	***	NS	**	**	**	***

***Significant at 0. 01 level; **significant at 0.05 level; *significant at 0.1 level, NSinsignificant.

(Table 2). These results are comparable to those observed in four fiber flax cultivars studied by Morrison and Akin (22). Temperature ($p < 0.05$) and pressure ($p < 0.1$) had a significant effect on the content of total sterols in flax wax; however, the flow rate effect was insignificant ($p < 0.1$). Similarly, the temperature and pressure ($p < 0.05$) showed a significant effect on the content of aldehydes in flax wax. In addition, 4 to 74 mg/100 g of aldehydes were extracted from flax, and these results concur with those of Morrison et al. (8).

The wax ester content in the wax of flax varied from 6 to 46 g/100 g, and temperature ($p < 0.01$), pressure ($p < 0.05$) and flow rate ($p < 0.1$) showed a significant effect on the extraction of wax esters from flax straw.

Analysis of Response Surfaces

Multiple regression analysis was performed to determine the regression coefficients of the model. Estimated coefficients of second-order response models generated from the statistical analysis of fatty acid, primary alcohol, alkane, sterol, aldehyde and wax ester yields are presented in Table 3. The coefficient of determination (R^2) and lack of fit values were higher for the wax yield, fatty acids, and wax esters. Based on canonical analysis of the second order polynomial model, the yield of wax from flax straw showed a maximum stationary point in three dimensional response surface plot (Fig. 1). The yield of wax from flax straw increased with an increase in pressure and temperature and reached a maximum at 378 bar and 75°C. In this study, the predicted yield at the stationary point was 1.27 g/100 g. Deswarte et al. (6), applied an experimental design (2^2 full factorial) on $SC\text{-}CO_2$ to determine the optimal temperature and pressure conditions for a maximum yield of the desired waxes from wheat straw, and the highest wax yield was obtained at 300 bar and 40°C. Thus, process conditions of $SC\text{-}CO_2$ extraction differ from one matrix to another, even for the same group of isolated compounds (12).

Temperature was found to be the most important factor affecting the yield of wax, with a highly significant ($p < 0.01$) negative quadratic effect (Table 2). Additionally, the interaction of temperature and pressure showed a negative and significant effect ($p < 0.01$). The density of $SC\text{-}CO_2$ is a function of pressure, and temperature plays a major role in determining the yield of extraction. An increase in pressure with a concomitant decrease in temperature allows greater contact between the solute and the solvent, leading

TABLE 3
Regression coefficients of predicted models for flax straw components and effects of process variables

		Main compounds					
Variable	Total yield	Fatty acids	Primary alcohol	Alkanes	Sterols	Aldehydes	Wax esters
$\beta 0$	−1.857854*	−634.731444***	−216.542243NS	68.439531NS	−106.374068NS	344.371337**	−82.670599NS
$\beta 1$	0.023878NS	12.697112***	3.054370NS	0.573621NS	1.605184NS	−4.457360NS	-0.110563NS
$\beta 2$	0.005016*	1.286477**	0.572633 NS	0.076712NS	0.169034NS	−1.270272***	0.347440**
$\beta 3$	0.038763*	7.805892**	4.579766*	−2.589900NS	3.306812*	−0.915009NS	1.119348NS
$\beta 11$	−0.000378**	−0.091933***	−0.022950 NS	−0.011377NS	−0.016910NS	0.044930**	−0.004799NS
$\beta 22$	0.000021NS	−0.004676NS	−0.003828 NS	0.000462NS	0.000917NS	0.004155NS	−0.001987NS
$\beta 33$	−0.000012***	−0.002517***	−0.000697*	−0.000445NS	−0.000619**	0.000600NS	−0.000252NS
$\beta 12$	0.000194NS	−0.006172NS	0.005178 NS	0.007811NS	0.002167NS	−0.023578NS	0.025722**
$\beta 13$	0.000034NS	0.011243**	0.002018 NS	0.003442NS	0.003530NS	0.012292***	−0.001002NS
$\beta 23$	−0.000426***	−0.075848***	−0.044319**	0.014377NS	−0.033854**	−0.011413NS	−0.014090*
Model	***	***	NS	*	*	*	***
Linear	***	***	NS	**	**	NS	***
Quadratic	***	***	*	NS	**	*	NS
Cross-product	NS	*	NS	NS	NS	**	*
R^2	0.9096	0.9486	0.7144	0.7642	0.7902	0.7711	0.8714
Lack of fit	0.2520	0.3028	0.0002	0.0234	0.0451	0.0010	0.0401

***Significant at 0. 01 level; **significant at 0.05 level; *significant at 0.1 level, NSinsignificant; 1, Flow rate; 2, Pressure and 3, Temperature.

to enhanced solute solubility and an increase in yield (23). The effect of temperature on solubility is complex in the SC-CO_2 system. Below the crossover pressure where the compressibility is larger, the solubility decreases with increasing temperature. At the pressure above the crossover pressure, the vapor-pressure effect dominates, hence solubility increases with temperature (24). In this study,

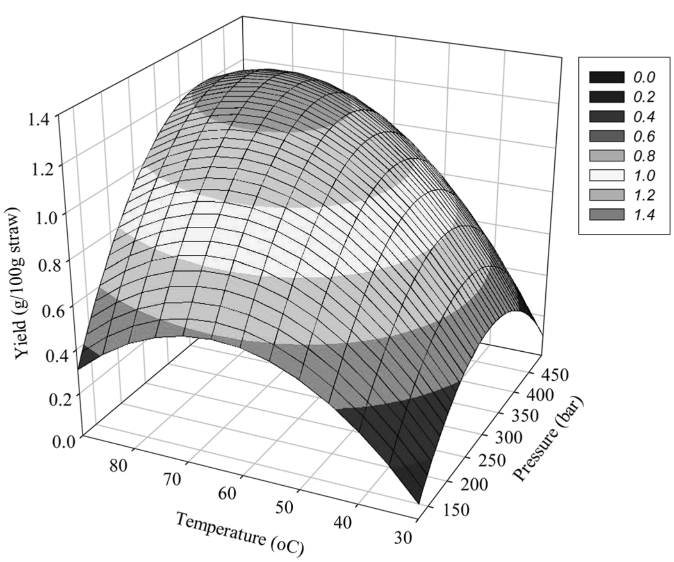

FIG. 1. Response surface showing the effect of extraction pressure and temperature on the yield of wax from flax straw.

the linear and quadratic effects were highly significant and influenced the wax yield of SC-CO_2 extraction; the value of R^2 and lack of fit were 0.91 and 0.25, respectively (Table 3).

To study the combined effect of temperature and pressure on the yield of fatty acids, primary alcohols, alkanes, sterols and aldehydes, contour plots were constructed using second order equations (Fig. 2). A higher R^2 value (0.94) than other counterparts (alcohols, alkanes, sterols, aldehydes, and wax esters) and lack of fit (0.30) were observed for fatty acids obtained from flax straw, and the linear and quadratic effects were highly significant (Table 3). In this study, the lower R^2 values of primary alcohol, alkane, sterol, aldehyde, and wax ester than fatty acids may be associated with their low amount in flax wax. The flow rate, pressure, and temperature showed a significant linear effect on fatty acid content. Additionally, quadratic and interaction effects of the flow rate and temperature were also highly significant ($p < 0.01$). A contour plot for the yield of fatty acid is shown in Fig. 2a, where the flow rate was fixed at 50 g/min. The amount of fatty acid extracted from flax wax was maximal at a flow rate of 57 g/min, a pressure of 374 bar and a temperature of 77°C. Under optimal conditions, a yield of 262 mg/100 g was predicted.

Linear and quadratic effects of temperature were significant for the extraction of primary alcohols from flax wax. Interaction effects of pressure and temperature were also significant for primary alcohols ($p < 0.05$). The effect of pressure and temperature on the yield of primary alcohols

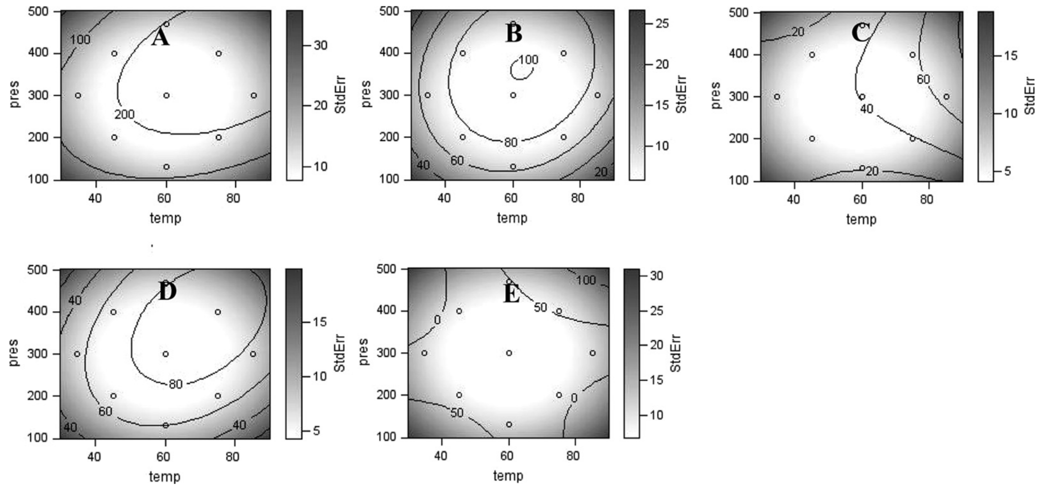

FIG. 2. Contour plots showing the effect of extraction temperature and pressure on the yield of main compounds from flax straw. (A), fatty acids; (B), fatty alcohols; (C), alkanes; (D), sterols and (E), aldehydes. Values were fixed at temperature 60°C, pressure 300 bar and flow rate 50 g/min.

at a fixed flow rate (50 g/min) is shown in Fig. 2b. In canonical analysis, all eigenvalues for primary alcohols were negative; thus, the stationary point was at a maximum. The predicted value for the maximum point was 101 g/100 g at a flow rate of 41 g/min, a pressure of 389 bar and a temperature of 63°C. According to De-Lucas et al. (25), optimal conditions for the extraction of long chain *n*-alcohols from sugarcane crude wax by SC-CO_2 included a pressure of 350 bar and a temperature of 100°C at 20% KOH saponification.

The stationary point for alkanes in flax wax was a saddle point, and the contour plot as a function of pressure and temperature at a fixed flow rate is shown in Fig. 2c. The linear, quadratic, and interaction effects of the flow rate, pressure, and temperature were insignificant for the extraction of alkanes (Table 3). Figure 2d shows the effect of temperature and pressure on the extraction of total sterols from flax wax. Linear and quadratic effects of temperature were significant for the extraction of sterol, and the interaction effects of temperature and pressure were significant ($p < 0.05$). The stationary point of sterol was at a maximum, and the highest predicted yield was 93 mg/100 g at a flow rate of 62 g/min, a pressure of 385 bar, and a temperature of 71°C. According to previous reports (26) the solubility of stigmasterol and beta-sitosterol, which were soild under pressure, were lower than or close to the minimum pressure used. The canonical analysis of aldehydes displayed a stationary saddle point. Linear effects of pressure and the interaction effects of temperature and flow rate were highly significant for the yield of aldehydes. A cross product effect was also significant ($p < 0.05$).

Linear effects of temperature and the interaction effects of the flow rate and pressure were significant for the extraction of wax esters ($p < 0.05$). The stationary point for the yield of wax esters was a saddle point and is shown in Fig. 3 as a function of temperature and pressure. The amount of wax esters extracted from flax increased with pressure and decreased with temperature. This may be due to the increase in density of SC-CO_2. As the density increases, the distance between molecules decreases and interactions between oil and CO_2 increase, leading to higher solubility in SC-CO_2 (27). As shown in Fig. 3 and Table 2, the effect of pressure and temperature is dominant in determining the rate of mass transfer and separation of wax esters from flax straw. Additionally, the chain length influences the solubility of compounds in SC-CO_2; compounds with shorter chains extracting faster than longer chain compounds (23).

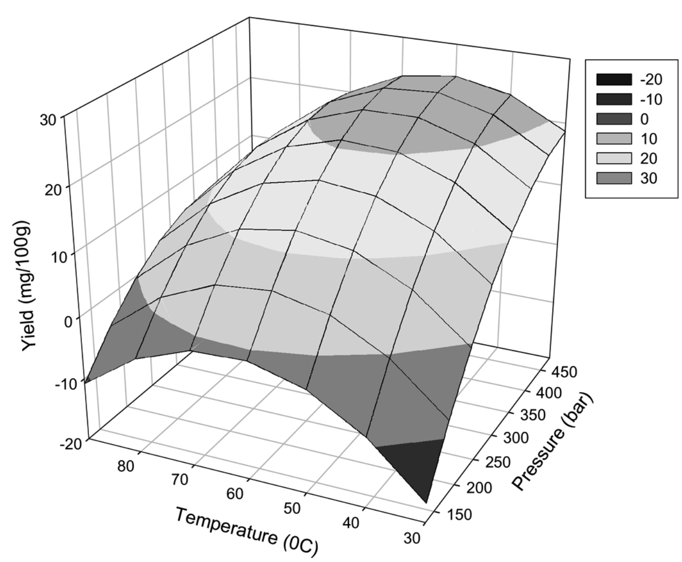

FIG. 3. Response surface showing the effect of extraction pressure and temperature on the yield of total wax esters from flax straw.

The results of this investigation revealed that the composition of wax derived from flax changed considerably with the flow rate, temperature, and pressure. Specific molecular properties, position of a functional group within a molecule, hydrogen bonding, solute-solute interactions, and solute-solid matrix interactions strongly interfere with the solubility of compounds in SC-CO_2. These factors may cause the observed solubility behavior in SC-CO_2; however, the effect of temperature is dominant in determining the rate of mass transfer and diffusion of wax from flax straw.

CONCLUSIONS

The successful use of SC-CO_2 extraction for the manipulation of lipid components (fatty acids, primary alcohols, alkanes, sterols, aldehydes, and wax esters) requires an understanding of their physical and chemical properties, as well as solubility behavior. Factors such as material size, extraction time, and moisture content also affect the efficiency, composition, and yield of SC-CO_2 extraction, but they were not included in this study. SC-CO_2 extraction of wax from flax straw was optimized, with the optimal yield obtained at a flow rate of 61.1 g/min, a pressure of 377.8 bar, and a temperature of 74.7°C. Temperature and pressure of SC-CO_2 showed significant effects (linear, quadratic, and interaction) on the extraction of wax from flax. A second order polynomial model developed for wax yield exhibited a high coefficient of determination and non significant values of lack of fit. Thus, the results show that wax yield from SC-CO_2 can be optimized to achieve considerable amounts of constituents, especially sterols, fatty alcohols, and wax esters.

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