TABLE I

Fatty Acid Composition of *Cuphea* Seed Oils

Species	Coll No.	Per cent fatty acid									
		8:0	10:0	12:0	14:0	16:0	18:0	18:1	18:2	18:3	20:0	20:1
Sect. *Brachyandra*												
C. calophylla C & S	839	Tr[a]	8.7	82.8	3.6	1.0	Tr	1.5	2.4	–	–	–
	847	–	0.2	76.5	7.6	4.1	0.5	3.9	6.5	0.7	–	–
	858	0.2	15.1	76.6	2.5	1.4	Tr	1.7	1.9	0.6	–	–
C. carthagenensis Macbr.	850	Tr	5.3	81.4	4.7	1.7	0.2	2.7	3.8	0.2	–	–
	915	0.5	15.8	60.1	7.8	2.4	0.7	5.9	6.7	0.1	–	–
Sect. *Cuphea*												
C. fruticosa Spreng.	916	–	Tr	Tr	0.1	16.8	0.4	12.8	67.2	–	2.0	0.7
Sect. *Euandra*												
C. diosmifolia St.-Hil.	866	Tr	Tr	64.0	31.3	1.8	0.4	1.5	1.0	–	–	–
C. glutinosa C & S	845	Tr	5.4	81.7	2.5	3.0	0.4	1.5	5.2	0.3	–	–
	911	0.6	26.1	59.1	3.8	1.3	0.2	2.8	5.7	0.4	–	–
C. linarioides C & S	840	Tr	0.2	3.2	3.1	17.7	2.1	11.6	62.1	–	–	–
C. linifolia Koehne	857	Tr	Tr	0.4	3.1	17.9	1.9	13.7	62.5	0.5	–	–
C. polymorphoides Koehne	913	Tr	7.4	80.1	3.6	2.0	0.1	2.5	4.3	–	–	–
C. pseudovaccinium St.-Hil.	895	0.5	3.3	68.8	8.0	5.5	1.7	6.5	2.9	2.8	–	–
	901	Tr	10.0	83.0	5.1	0.8	Tr	0.6	0.5	Tr	–	–
C. scelerophylla Koehne	883	Tr	Tr	59.7	27.6	5.3	0.2	2.1	5.1	Tr	–	–
C. thymoides C & S	841	Tr	0.5	55.8	7.0	9.4	1.7	7.9	17.4	0.3	–	–
Sect. *Heteranthus*												
C. tetrapetala Koehne	3528	–	0.4	32.4	51.0	7.0	0.9	1.5	6.4	0.4	0.1	–
Sect. *Melvilla*												
C. melvilla Lindl.	908	Tr	0.3	46.2	13.1	8.7	1.5	11.8	17.2	1.2	–	–
Sect. *Pseudocircaea*												
C. lutescens Koehne	864	Tr	0.1	76.3	19.3	1.6	Tr	1.1	1.2	0.4	–	–

[a]Tr = Trace, < 0.1%.

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♣Processing Characteristics and Oxidative Stability of Soybean Oil Extracted with Supercritical Carbon Dioxide at 50 C and 8,000 psi[1]

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ABSTRACT

The crude oil extracted from soy flakes with supercritical carbon dioxide ($SCCO_2$) was characterized for color, free fatty acid, phosphorus, neutral oil loss, unsaponifiable matter, tocopherol and iron content and compared to a commercial hexane-extracted sample of crude degummed oil. Characterization and processing studies indicate that $SCCO_2$ extraction yields a product comparable to a hexane-extracted degummed oil. However, hexane-extracted degummed soybean oils exhibit better oxidative stability because phosphatides, which are natural antioxidants, are essentially absent in $SCCO_2$-extracted oils.

[1] Presented at AOCS Meeting, Toronto, May, 1982.

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INTRODUCTION

Previous reports from this laboratory have shown that extraction of soy flakes with $SCCO_2$ at 50 C and 8,000 psi yields crude oil similar to degummed hexane-extracted oil (1,2). This report presents some properties and processing data for soybean oils obtained by $SCCO_2$ extraction under the aforementioned conditions.

EXPERIMENTAL

The $SCCO_2$ extraction methodology has been described previously (1,2). Soybeans were Tiger Brand certified seeds. AOCS Official Methods were used for the analyses (3). Processing (4) and oil evaluation methods (5) were described

TABLE I

Processing[a] and Properties of $SCCO_2$- and Hexane-Extracted Crude and Finished Soybean Oils

Crude oils	FFA %	Lovibond Color Y	Lovibond (5¼") R	Phosphorus ppm	Neutral oil Loss %	Iron, ppm	Unsaponifiables %	Tocopherols µg/g
$SCCO_2$-50 C 8,000 psi	0.30	70	8	1	0.6	0.3	0.66	1620
Hexane degummed	0.28	70	10	153	1.1	0.7	0.64	1020

Finished oils	Refining % excess 10% Lye	Color-Lovibond (5¼") Refined Y	Refined R	Bleached Y	Bleached R	Deodorized Y	Deodorized R	Flavor scores and significance[b] 0 Time	Flavor scores and significance[b] 4 days 60 C
CO_2	0.2	70	6	35	3.5	10	0.3	7.4 / 7.1 +	5.9 / 5.8 +
CO_2	0.5	70	6	35	4.0	10	0.3		
CO_2	0.05	35	7	35	3.5	6	0.3	7.1 / 7.4 +	6.3 / 6.3 +
CO_2	0.10	35	7	35	3.5	8	0.3		
Hexane	0.05	40	9	40	4.5	8	0.2	8.0 / 8.4 +	7.1 / 7.4 +
Hexane	0.10	40	8	40	3.5	8	0.2		

[a]Refining at 60 C, 5 min contact time, bleaching ½% activated clay 105 C, 15 min vacuum, deodorization 3 hr, 210 C, 1 mm Hg.

[b]+ denotes no statistical significance, oils contained .01% citric acid added on the cooling side of deodorization.

previously. Tocopherol was determined according to AOAC Method 43-092 (6). The crude, crude-degummed and lecithin samples were obtained from commercial sources. Oxidative stability of crude oils was determined according to Olcott and Einset (7).

RESULTS AND DISCUSSION

Processing and compositional data for $SCCO_2$- and hexane-extracted oils are given in Table I. The free fatty acid and unsaponifiable matter for $SCCO_2$- and hexane-extracted crude oil are nearly identical. Although the color of $SCCO_2$-extracted oil was about 2 red units lighter than the hexane-extracted oil, color removal at later stages of processing was virtually identical for both oil types. As reported previously, phosphatides show little solubility in $SCCO_2$ and therefore the product contains more neutral oil than a hexane-extracted crude degummed oil. Unpublished work has indicated that undefined parameters in the $SCCO_2$ process affect the tocopherol content of crude oils. Thus, the tocopherol content of the $SCCO_2$ extracted oil was somewhat higher than the hexane-extracted oil.

Portions of $SCCO_2$- and hexane-extracted oils were refined with 10% NaOH in excesses ranging from .05-0.5%. Color measurements taken after refining, bleaching and deodorization showed that the amount of excess refining lye has little effect on color removal.

Flavor evaluations showed that oils of good initial quality were obtained with flavor scores of 7-8 on a 10-point scale (10 = bland, 1 = extreme). Flavor scores after 4 days storage at 60 C indicated that normal deterioration had occurred during accelerated storage. That no significant differences were found between the initial or aged flavor scores of any of the oils suggests that the lye requirements for refining a $SCCO_2$-extracted crude oil are only slightly more than theoretical and less than required to refine

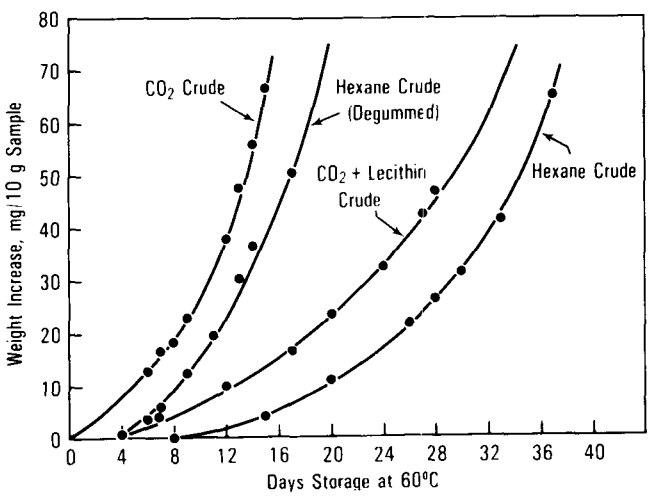

FIG. 1. Oxidative stability of $SCCO_2$- and hexane-extracted crude oils under Schall oven storage conditions: 10-g sample in 100-ml beaker. $SCCO_2$ crude + lecithin contained 2% commercial fluid unbleached lecithin.

conventionally extracted oils. Both hexane- and $SCCO_2$-extracted oils refined well with 10% NaOH at excesses of .05-.1%. These results indicate that $SCCO_2$ extraction yields a crude oil having properties very much like those of oil obtained by hexane extraction and degumming.

Published work has shown that the oxidative stability of soybean oil decreases with processing. Crude oil is the most stable, followed by degummed oil, with refined and bleached oil the least stable (8). The relative oxidative stability of $SCCO_2$ and hexane crude oils are shown in Figure 1. Despite the rather high levels of tocopherol found in $SCCO_2$-extracted oil, it is markedly less stable than a hexane-extracted crude oil. The role of phosphatides in

protecting crude oils from oxidation is not entirely clear, and it is not known whether they act as true antioxidants, as metal inactivators, or as synergists in conjunction with naturally occurring tocopherols. Nonetheless, results in Figure 1 show that degumming of hexane-extracted crude oil lowers oxidative stability and that addition of soy phosphatides to $SCCO_2$-extracted crude oil markedly improves oxidative stability. Although further work is required to elucidate the mechanism by which phosphatides protect crude oils, they may act as oxygen barrier at the oil/air interface and thus reduce the rate of oxygen uptake by the sample. However, it should be pointed out that a high level of tocopherol in the absence of phosphatides is not sufficient to protect crude $SCCO_2$-extracted oils and that they should not be stored for extended periods.

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Development of a Pilot Plant Process for the Preparation of a Soy Trypsin Inhibitor Concentrate[1]

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ABSTRACT

A pilot-plant procedure was developed to prepare a soy trypsin inhibitor (TI) concentrate in sufficient quantities to support a lifetime (2-yr) feeding trial in which diets containing varying amounts of TI would be fed to rats to assess the physiological effects on the pancreas and other organs. Starting with water dispersions of commercial defatted soy flour, separation of TI (MW<21,500) from non-TI protein (MW 180,000-350,000) by virtue of their MW difference was attempted using ultrafiltration techniques but was not successful. However, good separation was obtained when selective acid precipitation coupled with "salting in" of the TI with 0.1 N sodium chloride was employed. Low MW components were separated successfully by ultrafiltration using a 1,000 MW cutoff membrane. The final soy TI concentrate obtained by freeze drying exhibited a 9-fold increase in TI activity.

INTRODUCTION

Naturally occurring proteinase inhibitors are substances that exist in a wide variety of food crops and other foods, such as soybeans, potatoes, lima beans and egg whites (1,2). Many short-term animal feeding studies have shown that raw soybean meal and purified soybean trypsin inhibitors (TI) inhibit growth and enlarge the pancreas in certain monogastric animals (3,4). Very limited information (5) is available on the effect of various levels of TI in the diets of rats over a life-time (2-yr) feeding trial because of the considerable costs generated over the 2-yr period and the general unavailability of sufficient quantities of TI concentrate from various food sources. Previous work by the USDA involved processing 24,000 lb of potatoes at two locations (ERRC and NRRC) to produce 34 lb of a potato TI concentrate (6). It appeared desirable to test, at the same time, the effects of TI isolated from soybeans. The purpose of this study was to develop pilot-plant procedures to prepare a sufficient quantity of TI concentrate from soy to sustain a 2-yr rat feeding trial.

MATERIALS AND METHODS

Commercial defatted soy flour Nutrisoy 7B (Archer Daniels Midland, Decatur, Illinois) was used as the starting material in all experiments. The commercial process uses a mild heat treatment and results in minimal denaturation of protein as evidenced by the nitrogen solubility index of 82.6 (Table I). Most of the TI activity of the raw beans is retained in the soy flour. Chemicals used in cleaning and sanitizing process equipment surfaces were Foam-nox, Status, AC-101 and XY-12 (Klenzade Division, Economics Laboratories, St. Paul, Minnesota). Ultrafiltration modules were cleaned with Ultrazyme (Osmonics Inc., Minnetonka, Minnesota).

Trypsin inhibitor was determined by the method of Hamerstrand et al. (7), which evolved primarily from the work of Kakade et al. (8). Total solids were determined by evaporation. Bacterial plate counts were made on standard methods agar (Baltimore Bacteriological Laboratories, Baltimore, Maryland) and incubated 3 days at 28 C. Nitrogen, ash and nitrogen solubility index were determined by official AOCS methods (9).

Equipment and Procedure

Acid precipitation of protein curd. Defatted soy flour (50 lb) was suspended in 10 to 15 parts of water in a 130-gallon stainless steel agitated tank fitted with an internal coil for heating or cooling. Slurry temperature was varied from 7 C to 49 C. TI was "salted in" by the addition of sodium chloride or magnesium chloride to 0.1 N. Protein curd was precipitated by the addition of dilute sulfuric acid (pH range 3.7 to 5.0). The protein curd was separated from

TABLE I

Composition and Microbial Population of Commercial Defatted Soy Flour

Component		Plate count per gram	
Moisture, %	6.6	Aerobic bacteria	37,500
Ash, %	6.0	Anaerobic bacteria	1,400
Nitrogen, %	8.0	Molds	120
Protein (N × 6.25), %	49.8	Yeast	0
NSI	82.6		
Trypsin inhibitor mg/g	27.6		

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[1]Presented at the AOCS Annual Meeting, Dallas, May 1984.