Food Chemistry **49** (1994) 39–44

Alkanol–ammonia–water/hexane extraction of flaxseed

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(Received 6 November 1992; revised version received and accepted 15 March 1993)

A two-phase solvent-extraction system consisting of alkanol–ammonia–water and hexane was employed for the simultaneous extraction of oil and detoxification of flaxseed meal. Alkanols used were methanol, ethanol, and isopropanol. The two-phase solvent extraction consisting of methanol–ammonia–water/hexane resulted in the highest protein enrichment of the meal. However, the process did not change the content of non-protein nitrogenous compounds to any great extent. The methanol–ammonia–water/hexane extraction system, in addition to reducing the content of cyanogenic glycosides, performed well in reducing the content of phenolic acids, condensed tannins, and soluble sugars of the meal. However, phytic acid was not removed from the meals (on a protein basis). The oil recovered from the non-polar phase of the solvent system had an unaltered fatty-acid composition.

INTRODUCTION

Flaxseed (*Linum usitatissimum* L.) is mainly grown for its oil, which is generally intended for industrial applications. It ranks sixth among the major oilseed crops in the world (Anon., 1989), of which Canada is the largest producer, and it contributes 28% to the total global production. The oil from flax or linseed, as it is most commonly termed, is obtained by solvent extraction or expeller pressing and is used primarily as a drying oil in paints, varnishes, and other industrial and/or pharmaceutical applications. It contains a relatively high content of α-linolenic acid, from 12–72% of the total fatty acids (Dorrel, 1970; Anderson, 1971; Patterson, 1989). The use of linseed oil to enrich the polyunsaturated fatty-acid content of animal tissues has been investigated (Holub, 1990; Jiang *et al.*, 1991). It has also been demonstrated that copper-catalysed hydrogenation of linseed oil is suitable for the production of an edible product for margarine blends (Anderson, 1971). However, the development of low-linoleate flaxseed varieties may allow the production of food-grade linseed oil (Dorrel, 1972; Prentice, 1992).

Flaxseed has been used in a variety of speciality food products (Dybing & Lay, 1981), where a small amount of seed is used. However, the deoiled flax meal is mainly employed as an animal-feed component. The crude protein content of flaxseed meal ranges from 36 to 45% (Singh, 1979; Madhusudhan & Singh, 1983; Bhatty & Cherdkiatgumchai, 1990), and in this respect it is similar to canola. Studies have shown that flaxseed meal can improve milk production, carcass grade, and the finish and appearance of the products (Peterson, 1958). The upper limit for the incorporation of flax meal in different feed formulations is 14% for dairy animals (Singh, 1979), 25% for swine (Peterson, 1958; McDonald *et al.*, 1966), and 5% for poultry (McDonald *et al.*, 1966). However, Ratnayake *et al.* (1992) have shown that even 40% incorporation of flaxseed meal in the diets of rats did not result in any adverse effects on their growth rate.

Several researchers (Madusudhan & Singh, 1983, 1985*a,b*; Dev & Quensel, 1986, 1989) have studied the possible use of flax meal in food products. The presence of antinutrients is a major factor that limits the use of flax meal in feed and food formulations. Cyanogenic glycosides (Smith *et al.*, 1980; Conn, 1981) and an anti-vitamin B_6, referred to as linatine (Klostermann *et al.*, 1967), have been reported to be the major antinutrients in linseed meal. Several methods have been described to detoxify the meal by employing heat and moisture treatments (Madhusudhan & Singh, 1985*a*); however, these processes adversely affected the quality of the meal. The development of a novel method for the removal of antinutrients from flaxseed without adversely affecting its nutritional quality or functional properties is therefore required.

The objective of the work reported in this paper was to study the effects of a two-phase solvent-extraction processing method on the simultaneous extraction of

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Food Chemistry 0308-8146/93/$06.00 © 1993 Elsevier Science Publishers Ltd, England. Printed in Great Britain

oil and detoxification of meal from flaxseed. The contents of nutritional and antinutritional components of the meals, as affected by this process, were then determined.

MATERIALS AND METHODS

Seeds and meals of flax were obtained from either Omega Nutrition (Vancouver, British Columbia, Canada) or through the Flax Council of Canada (Winnipeg, Manitoba, Canada). Seeds were first crushed by using a Moulinex coffee grinder. Hexane-extracted meals were prepared by blending seed or meal (75 g) with 500 ml of hexane in a Waring blender for 2 min at low speed (approximately 10 000 r/min). Other treatments were carried out on the crushed seeds by first blending meal (75 g) with 500 ml of 95% (v/v) methanol, ethanol, or isopropanol with or without 10% dissolved (w/w) ammonia for 2 min at 10 000 r/min, this being followed by a 15-min quiescent period, and then the addition of 500 ml of hexane to the mixture and subsequently blending it for 2 min at 10 000 r/min. Treated meal was next separated by vacuum filtration on Whatman No. 41 filter paper, rinsed three times with a total of 125 ml of alkanol, and dried overnight at 40°C in vacuum oven. The alkanol phase was re-extracted three times with hexane at an alkanol-to-hexane ratio of 2:1 (v/v) to recover any residual oil. The alkanol phase was evaporated under vacuum to recover dissolved solids. The meal was further defatted with hexane by using a Soxhlet apparatus and dried as before. Dried meals were stored in airtight plastic bags at room temperature.

Proximate composition

The moisture content of meals was determined according to the method of AOAC (1990). The content of crude proteins was calculated from Kjeldahl nitrogen values (N% × 6·25). The ash content of the samples was determined by the AOAC (1990) procedure. The content of non-protein nitrogen was determined by the method of Bhatty and Finlayson (1973) as modified by Naczk *et al.* (1985). Meal (1 g) was shaken with 40 ml of 10% (w/w) trichloroacetic acid (TCA) solution at 20°C for 1 h by using a Burrel wrist action shaker (Pittsburgh, PA, USA). The insoluble residue was removed by centrifugation at 5000*g* for 10 min. The residue was extracted three times with 15 ml of 10% (w/w) TCA solution. The supernatants were collected as before and were combined and made up to 100 ml with distilled water. An aliquot was taken for the determination of soluble nitrogen by the Kjeldahl procedure (AOAC, 1990).

Phenolic compounds

The free, esterified, and insoluble bound phenolic acids of flaxseed meals were isolated according to the procedure described by Krygier *et al.* (1982). Meals (2 g) were extracted six times with 40 ml of methanol/acetone/water (7:7:6, v/v/v) at room temperature, a Polytron homogenizer was used for 1 min at 10 000 r/min. After each extraction, samples were centrifuged for 15 min at 5000*g*, and supernatants were collected. Combined supernatants were evaporated at 30°C under vacuum to 40 ml, and the pH of the mixture was adjusted to 2 by using 6N HCl. Samples were centrifuged at 5000*g* and supernatants were extracted six times with diethyl ether/ethyl acetate mixture (1:1, v/v) at a supernatant-to-solvent ratio of 1:1 (v/v). The ether extracts were combined and evaporated to dryness at 30°C under vacuum. The extracted phenolic acids contained free phenolic acids (FPA). The aqueous layer remaining after the extraction of free phenolic acids and the precipitate after centrifugation were then treated with 30 ml of 4N NaOH under nitrogen for 4 h at room temperature to release esterified phenolic acids. The resultant hydrolysate was acidified to pH 2 by using 6N HCl and extracted into a diethyl ether/ethyl acetate mixture (1:1, v/v) and dried, as before, to yield esterified phenolic acids (EPA). The remaining meal after the extraction of free and esterified phenolic acids was treated with 20 ml of 4N NaOH under nitrogen at room temperature and then acidified with 6N HCl to pH 2 and centrifuged at 5000*g* for 15 min. The supernatants were extracted six times with the diethyl ether/ethyl acetate mixture as before, and extracts were evaporated to dryness under vacuum to obtain insoluble bound phenolic acids (IBPA). The dried phenolic acids (FPA, EPA, and IBPA) were dissolved in methanol separately, and the content of phenolic acids in each was determined colorimetrically by using Folin–Denis reagent as described by Swain and Hillis (1959). The total content of phenolic acids was calculated as the sum of free, esterified, and insoluble bound fractions and expressed as mg ferulic acid equivalents per 100 g meal. The content of condensed tannins of the meals was estimated by the method described by Naczk and Shahidi (1989).

Phytic acid

Phytic acid was extracted from prepared meals according to the method of Tongkonchitr *et al.* (1981) as modified by Naczk *et al.* (1986). A 2·0-g meal sample was extracted with 40 ml of 1·2% HCl containing 10% Na_2SO_4 for 2 h by means of a wrist-action shaker. The slurry was centrifuged for 20 min at 5000*g*. The supernatant (5 ml) was mixed with 5 ml of distilled water and 6 ml of 0·4% $FeCl_3.6H_2O$ in 0·07N HCl solution. The mixture was heated in a boiling-water bath for 45 min and then cooled to room temperature. The resulting ferric phytate precipitate was collected by centrifugation at 5000*g* for 15 min, and the supernatant was discarded. The precipitate was mixed thoroughly with 5 ml of 4% Na_2SO_4 in 0·07N HCl, and the mixture was centrifuged again. The recovered ferric phytate was digested by using 6 ml of a 1:1 (v/v) mixture of

Table 1. The effect of different alkanols on the mass balance in the two-phase solvent-extraction process of linseed[a]

Alkanol	Water in alkanol (% v/v)	Ammonia in alkanol–water (% w/w)	% Yield on dry basis			Loss %
			Meal	Oil	Gums	
Methanol	—	—	46·7 ± 1·0	45·9 ± 1·9	5·2 ± 0·1	2·0
	—	10	47·6 ± 2·0	46·6 ± 0·8	4·8 ± 0·7	1·0
	5	—	46·7 ± 2·0	48·6 ± 0·5	4·8 ± 0·1	2·0
	5	10	46·4 ± 2·0	47·1 ± 0·1	5·7 ± 0·1	0·9
	5[b]	10	74·2 ± 2·0	14·3 ± 1·0	6·9 ± 0·2	4·6
Ethanol	5	10	48·1 ± 1·0	46·8 ± 1·8	4·2 ± 0·1	0·9
Isopropanol	5	10	50·0 ± 3·0	48·8 ± 0·5	No phase separation	1·2

[a] Hexane extraction of linseed yielded 48·9% meal, 49·2% oil, and 1·9% loss on a dry basis.
[b] A commercial meal was used in this study. It contained 82·5% solids, 15·1% oil, on a dry basis, and 2·4% loss.

concentrated H_2SO_4 and concentrated HNO_3. A 10-ml portion of distilled water was added to the warm digest, and the solution was heated in a boiling-water bath for 30 min to destroy pyrophosphate; the mixture was then diluted with distilled water to 100 ml. The phytate phosphorus of the extracts was determined according to the method of Nahapetian and Bassiri (1979). To 1 ml diluted digest, 4 ml of distilled water, 3 ml of 1·5N H_2SO_4, 0·4 ml of 10% $(NH_4)_6Mo_7O_{24}.4H_2O$, and 0·4 ml of 2% ascorbic acid were added and mixed. The solution was allowed to stand for 20 min, and absorbance was measured at 660 nm. The content of phosphorus in the mixture was calculated by using KH_2PO_4 as standard. The phytic acid content (%) in meals was calculated by multiplying the content of their phosphorus by a factor of 3·55 (derived from the empirical formula $C_6P_6H_{18}O_{24}$ for phytic acid).

Total soluble sugars

The total content of soluble sugars in the meals was determined by the method of Finley and Fellers (1973), involving the use of the Anthrone method (Carrol *et al.*, 1965). Results were expressed as sucrose equivalents.

Fatty-acid composition

The fatty-acid composition of flax oil recovered from the hexane phase was determined by using a gas-chromatographic procedure. Details have been described elsewhere (Shahidi & Synowiecki (1991).

Replications

All experiments and/or measurements reported in this study were replicated from three to six times. In each case, the mean value ± standard deviation was calculated.

RESULTS AND DISCUSSION

The amount of oil and meal recovered from flaxseed was 46–50% and 46–49%, respectively, and this depended largely on the solvent-extraction system employed (Table 1). A total of 4·8–5·7% of solids was extracted into the polar phase in this process. The use of methanol was emphasized in this study, since its efficiency in the removal of glucosinolates (Shahidi & Gabon, 1988, 1989, 1990) and phenolic compounds (Shahidi & Naczk, 1988, 1989; Naczk & Shahidi, 1989) from canola and rapeseed was superior to that of other alkanols. In addition, it was found to remove cyanogenic glycosides from flaxseed effectively (Wanasundara *et al.*, 1992). The content of crude protein in treated meals increased by 5·3–13·0%, depending on the treatment employed (Table 2). Commercial linseed meal had a slightly lower crude-protein content than its laboratory-prepared counterpart. Methanol alone had little effect on the content of proteins of the meal. The

Table 2. The effect of solvent extraction on contents of crude protein, ash, and non-protein nitrogen of linseed meal

Solvent	Crude protein (%N × 6·25)	Ash (%)	Non-protein nitrogen (% of total nitrogen)
Laboratory-prepared meal			
Hexane only	42·9 ± 0·3	5·57 ± 0·15	11·0 ± 0·5
Methanol/hexane	43·5 ± 0·7	5·93 ± 0·13	9·8 ± 0·8
Methanol–water/hexane	46·8 ± 0·3	5·80 ± 0·11	10·0 ± 0·9
Methanol–ammonia/hexane	46·0 ± 0·1	6·02 ± 0·10	10·3 ± 0·6
Methanol–ammonia–water/hexane	48·6 ± 0·3	6·03 ± 0·07	11·7 ± 0·3
Ethanol–ammonia–water/hexane	46·3 ± 0·2	5·97 ± 0·17	11·8 ± 0·2
Isopropanol–ammonia–water/hexane	46·0 ± 0·4	5·62 ± 0·02	9·5 ± 0·2
Commercial meal			
Hexane only	41·2 ± 0·5	6·17 ± 0·02	9·2 ± 0·1
Methanol–ammonia–water/hexane	47·6 ± 0·2	6·70 ± 0·30	9·6 ± 0·2

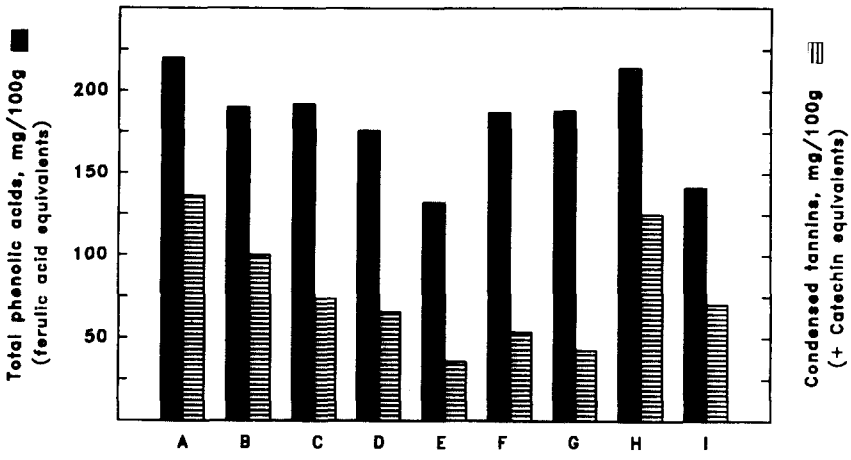

Fig. 1. Effect of solvent extraction on content of total phenolic acids and condensed tannins of linseed meal. A, hexane-extracted; B, methanol/hexane-extracted; C, methanol–water/hexane-extracted; D, methanol–ammonia/hexane-extracted; E, methanol–ammonia–water/hexane-extracted; F, ethanol–ammonia–water/hexane-extracted; G, isopropanol–ammonia–water/hexane-extracted; H, hexane-extracted commercial meal; I, methanol–ammonia–water/hexane-extracted commercial meal.

presence of ammonia or water in the solvent system increased the protein content of the meal. The largest increase was achieved when the methanol–ammonia–water system was employed. The ash content of meals increased by approximately 8% after solvent extraction (Table 2). The increase in crude-protein and ash contents may be primarily due to a concentration effect brought about by the removal of polar matters into the alkanol phase. However, the non-protein-nitrogen content of the meals did not change during the process, whereas Shahidi *et al.* (1988) reported that the non-protein-nitrogen content of canola was reduced by about 50% when methanol–ammonia–water/hexane extraction was employed.

The effect of the two-phase solvent-extraction system on the content of cyanogenic glycosides of flaxseed meal has previously been reported (Wanasundara *et al.*, 1992). The methanol–ammonia–water/hexane system was capable of removing cyanogenic glycosides of flaxseed meal by an order of magnitude under the optimum processing conditions.

The total content of phenolic acids (Fig. 1) of linseed meal was about 0·22% (220 mg/100 g), on a dry-weight basis. The content of phenolic acids reported by Kozlowska *et al.* (1983) and Dabrowski and Sosulski (1984) ranged between 60·4 and 81·2 mg/100 g of hull-free meals, respectively. Ferulic acid was the predominant phenolic acid present in flaxseed (Dabrowski & Sosulski, 1984). The presence of hulls in the meals in this study resulted in a higher content of phenolic acids than the contents reported in the literature. However, the content of phenolic acids in flax meal is much lower than that in canola meals. Methanol in combination with ammonia and water was the most effective system for the removal of total phenolic acids (approximately 48%). Existing differences in the microstructure of flaxseed as compared with that of canola may be responsible for the smaller extent of the removal of phenolic acids from flax than that of canola (Naczk & Shahidi, 1989).

The content of condensed tannins of flax, as affected by different solvent-extraction systems, and expressed

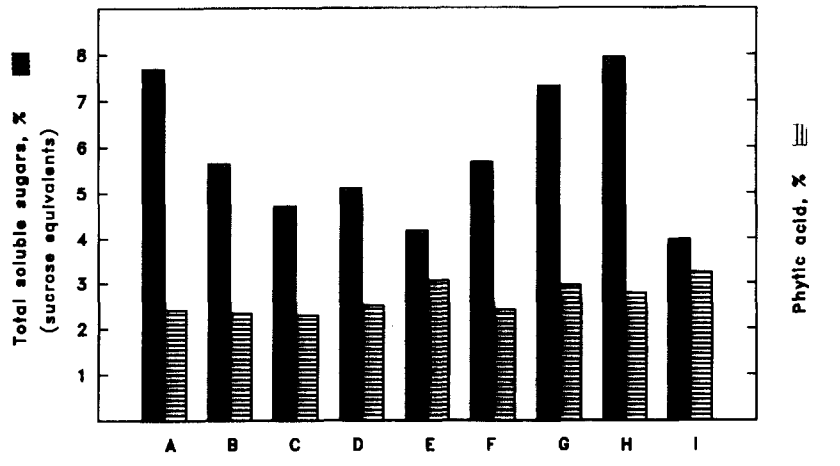

Fig. 2. Effect of solvent extraction on content of total soluble sugars and phytic acid of linseed meal. A, hexane-extracted; B, methanol/hexane-extracted; C, methanol–water/hexane-extracted; D, methanol–ammonia/hexane-extracted; E, methanol–ammonia–water/hexane-extracted; F, ethanol–ammonia–water/hexane-extracted; G, isopropanol–ammonia–water/hexane-extracted; H, hexane-extracted commercial meal; I, methanol–ammonia–water/hexane-extracted commercial meal.

Table 3. Fatty-acid composition (w/w, %) of linseed oil prepared by hexane and methanol–ammonia–water/hexane extractions

Fatty acid	Hexane extracted	Methanol–ammonia–water/ hexane extracted
C14:0	0·20 ± 0·07	0·25 ± 0·05
C16:0	5·05 ± 0·26	4·83 ± 0·20
C18:0	4·54 ± 0·75	3·64 ± 0·07
C20:0	0·33 ± 0·01	0·33 ± 0·01
C22:0	0·15 ± 0·03	0·21 ± 0·01
C16:1	0·10 ± 0·00	0·13 ± 0·01
C18:1	14·80 ± 0·10	14·50 ± 0·10
C18:2	17·50 ± 0·10	17·10 ± 0·10
C18:3n-3	55·60 ± 0·30	54·40 ± 0·90
C18:4n-3	0·17 ± 0·00	0·58 ± 0·02
C20:2	0·16 ± 0·05	0·17 ± 0·06
C20:3	0·10 ± 0·00	0·09 ± 0·00

as (+)-catechin equivalents, is presented in Fig. 1. Approximately 136 mg/100 g of condensed tannins were present in the meal, on a dry-weight basis. The two-phase extraction system reduced the tannin content of meals by 26–74%. The presence of ammonia in the extraction system increased the removal of tannins; thus methanol–ammonia–water/hexane and isopropanol–ammonia–water/hexane systems reduced the tannin content of the meals by 74% and 68%, respectively. A similar reduction in the condensed tannins of canola due to methanol–ammonia–water/hexane extraction was reported by Shahidi and Naczk (1989). The removal of phenolic acids and condensed tannins from oilseed meals is considered important since they are known to influence the formation of off-flavours and dark colours in the meals and may also participate in the binding of minerals and vitamins.

The content of phytic acid in resultant meals was 2·4–3·2%, on a dry-weight basis (Fig. 2). None of the two-phase solvent-extraction systems was able to reduce the content of phytic acid in linseed meal, and indeed an increase in their content was noticed. It is well established that phytic acid in oilseeds is present in association with storage proteins (Thompson, 1990). In the present study, the removal of polar substances from the meals resulted in the concentration of proteins, and a similar enhancement in the content of phytic acid occurred.

A decrease in the content of total soluble sugars due to the extraction of linseed with alkanols was observed (Fig. 2). Methanol–ammonia–water/hexane-extraction reduced the content of soluble sugars by approximately 46%. Other extractions, except isopropanol–ammonia–water/hexane, which had little effect, removed from 26–39% of the total soluble sugars present in the meal. The application of the methanol–ammonia–water/hexane system was effective in the removal of flatulence-causing sugars from commercial soybean meal (Myhara *et al.*, 1989). In canola, the dominant sugar, sucrose was removed by 78–89%; however, higher-molecular-weight sugars, such as raffinose and stachyose, were removed less effectively (Naczk & Shahidi, 1990). Literature on the composition of sugars of linseed meal is scarce, and further studies in this area are in progress.

The fatty-acid composition of linseed oil recovered from the hexane phase of methanol–ammonia–water treatment is presented in Table 3. The two-phase solvent extraction did not alter the fatty-acid composition of the oil to any great extent. The removal of phospholipids by methanol–ammonia–water may have been responsible for the small differences observed. Shahidi *et al.* (1988) reported that approximately 10% of the total phosphorous was removed by methanol–ammonia–water extraction of canola.

In conclusion, it has been demonstrated that the methanol–ammonia–water/hexane system was superior to other solvent mixtures in reducing the content of phenolic acids, condensed tannins, and soluble sugars of flaxseed meal. The removal of cyanogenic glycosides (Wanasundara *et al.*, 1992), phenolics, and flatulence-causing sugars by the above-mentioned systems is expected to improve the nutritional and sensory quality of the resultant meals. The efficiency of the removal of the latter compounds by the solvent mixtures employed was in the order of methanol–ammonia–water ≫ ethanol–ammonia–water > isopropanol–ammonia–water. The improved quality of flax meal so prepared provides a mean for the better utilization of meals in animal-feed and possibly human-food formulations.

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