Compositional Analysis of Laboratory-Prepared and Commercial Samples of Linseed Meal and of Hull Isolated from Flax

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Six laboratory-prepared (LM) and four commercially-obtained (CM) samples of linseed meal were analyzed for eleven proximate components, ten mineral elements, monosaccharides, amino acids, and seven vitamins (two samples only). Analysis of variance of LM data showed location had a greater influence on meal composition than did cultivar. LM and CM had similar composition, except for protein, total carbohydrates, acid-detergent fiber and lignin. Hull separated by a liquid cyclone process formed 37.5% of the seed and contained less than 1% oil, 20% protein and 32.9% total monosaccharides. Xylose and arabinose were the major sugars. Meal absorbed 8-fold, and the hull 13-fold their weights of water (water-hydration capacity), compared to less than 2-fold by similar fractions of canola (rapeseed) and soybean. Viscosities of aqueous extracts of hull were stable for 30 min at 25°C, and were concentration-dependent.

Flax (*Linum usitatissimum* L.) is grown in Canada essentially for industrial (linseed) oil. A major portion of the Canadian flax is exported as raw seed to Western Europe, and very little is crushed domestically to yield linseed meal, the by-product of the flax crushing industry. However, this may change in the near future based on the availability of edible flax oil containing reduced levels of α-linolenic acid, which will provide a major impetus to the Canadian oilseed crushing industry.

Linseed meal is largely used in livestock feeds, particularly for ruminants. Expeller meal containing about 3.5% crude fat is a popular ingredient in calf feed formulations in the United Kingdom. The proximate composition of linseed meal is not markedly different (except for fiber) than soybean meal, although its energy and apparent protein digestibilities, determined by mouse-feeding, were lower than some other oilseed meals (1). The use of linseed meal in poultry feeds is limited due to the presence of a vitamin B6 antagonist (2), identified as (N-ν-L-glutamyl)-amino-D-proline (3). The deleterious effects of the antagonist (linatine) may be partially alleviated by supplementing the meal with vitamin B6. Linseed meal may also contain other anti-nutritional factors, particularly cyanogenic glucosides (linamarin and methyllinamarin), the precursor of hydrocyanic or prussic acid (4). An earlier study (5) reported a goitrogenic effect of linseed meal in sheep due to thiocyanate produced on detoxification of the cyanogenic glucoside by liver. Linseed meal is not suitable as a sole source of protein for swine due to deficiency of essential amino acids lysine and methionine. On the other hand, many beneficial effects in feeding linseed meal to livestock have been associated with its mucilage content (4). Very little linseed meal is used in human foods, except in specialty foods and breakfast cereals (6).

Few comprehensive studies have been published on the composition of linseed meal, although proximate and mineral composition has often been reported (1,6–10). Most data reported in the literature on linseed meal composition are limited and not always comparable due to use, in some cases, of individual samples of unknown origin, and also older and less reliable methods of analysis. As far as the authors are aware, the last article on linseed meal appeared thirty years ago (4). A recent monograph on oilseeds (11) treated linseed meal only perfunctorily, due to limited information available in the literature.

The present study was conducted to obtain comparative information on the composition of laboratory-prepared and commercially-obtained samples of linseed meal analyzed under identical conditions. In addition, composition and water-holding properties of flax hull, obtained by a liquid cyclone process, were determined and compared to those of soybean and canola (rapeseed) hulls isolated under identical conditions. The objective was to determine the range and level of various components of linseed meal for its market potential in feed and food applications.

MATERIALS AND METHODS

Samples. Three licensed cultivars of flax (*Linus usitatissimum* L.), NorLin, NorMan and McGregor, each grown at two locations (Saskatoon, SK, and Morden, MB) in the 1987 Flax Cooperative Test were used in the study. The samples grown at Morden were supplied by Dr. E. Kenaschuk, Agriculture Canada Research Station, Morden, and those grown at Saskatoon by Dr. G. Rowland of this department. Four samples of commercial linseed meal (cultivar unknown) were obtained from Omega Nutrition, Vancouver, BC; Alberta Linseed, Medicine Hat, AB; ADM-Agri Industri, Windsor, ON; and Cargill, Riverside, ND (USA). One sample each of canola, cv. Westar (*Brassica napus* L.) and of soybean (*Glycine* max L.) Merr. were obtained locally.

Meal analysis. The flax was defatted and crushed to a meal by shaking with petroleum ether (b.p. 35–60°C) in a Swedish ball mill for 2–3 hr. After filtration, the meal was air dried and ground in a micro hammer mill to pass 0.5 mm screen. The commercial meals were defatted by stirring with petroleum ether for 30 min, filtered, air dried and ground as before.

Residual oil in the meals was determined by Goldfisch extraction for 5 hr (12). Moisture, ash, protein (N × 6.25), acid detergent fiber and lignin contents were determined by the AOAC methods (13).

Cellulose content was obtained from the residue dissolved in 72% sulfuric acid during lignin determination (14). Total dietary fiber was determined using the Sigma kit based on the procedure of Prosky *et al.* (15). Total carbohydrate content was determined with phenol-sulfuric acid, and raffinose was used as a standard (16). Starch was determined by a procedure described by Fleming and Reichert (17). Pentosan content was determined colorimetrically, using ferric chloride-orcinol reaction (18).

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Phytic acid was determined by an anion-exchange method followed by colorimetric determination of the total phosphorus (19). Mineral composition was determined after sequential hydrolysis of the meals with nitric, perchloric and hydrochloric acids, with an ICP spectrophotometer. Monosaccharides were determined after acid hydrolysis of the meals followed by reduction and acetylation (20). The alditol acetates were separated by gas-liquid chromatography operated under the following conditions: fused silica column, SP 2330 (Supelco, Oakville, ON); injection port and detector temperatures, 250 and 300°C, respectively; temperature program, 170°C to 230°C at 8°C/min; carrier gas (nitrogen) flow rate adjusted to complete the run in about 13 min; internal standard, *myo*-inositol; and integration system, Hewlett-Packard 3385A. Response factors were calculated for each sugar with authentic samples.

For amino acid analysis, the meals were hydrolyzed with excess of 5.7 M hydrochloric acid for 22 hr at 110°C. After filtration and evaporation the residues were taken up in the diluting buffer (pH 2.2). Amino acid composition was determined on a Terochem 911 analyzer. Cystine and methionine were determined after performic acid hydrolysis. Tryptophan was determined after barium hydroxide hydrolysis (21) on a Beckman 119 amino acid analyzer. Vitamin composition was determined by Diversified Research Laboratories, Toronto, using methods described in the literature (13,22,23).

Separation of flax into flour and hull fractions. Samples of flax (cv NorMan), soybean and canola (for comparison) were defatted and separated into flour and hull fractions by a liquid cyclone process (24). Moisture, residual oil, protein, and monosaccharide contents of the hull were determined as described above. Galacturonic acid was determined as described by Ahmed and Labavitch (25). Water-holding capacity of the meal, flour and hull fractions were determined according to the AACC procedure (26). For viscosity determination, different weights of hull were extracted with 15 ml of water or other solvents for 1 hr in a Udy shaker. The extract was centrifuged at 1000 × *g* for 20 min, and the viscosity of the supernatant solution was determined at 20°C, with an Ubbelohde viscometer.

The data, except for vitamins, are means of at least duplicate determinations and expressed on oil-free and moisture-free basis.

RESULTS AND DISCUSSION

Linseed meal. On receipt in the laboratory, the four commercial meals (CM) contained 1.1–16.2% residual oil. Each meal thus seemed to have been prepared under different conditions of oil extraction. Details of these conditions were not available. Furthermore, the meals were of different color and texture. Each meal was further defatted in the laboratory to obtain a residual oil content of 1–3%, which was in the range obtained for the laboratory-prepared meals (LM).

To determine the effect of location, cultivar and location-cultivar interaction on meal components, data obtained from LM were analyzed by analysis of variance. A similar analysis could not be conducted on data obtained from CM, as cultivar source and growth location were not known. Table 1 shows the F ratios and their level of significance for the location, cultivar and location-cultivar interaction. The growth location had a major effect on the meal composition. Only few cultivar effects (ash and phytic acid) and location-cultivar interactions (ash, phytic acid and pentosans) were statistically significant. The cultivar difference in phytic acid was due to low total phosphorus (P) content of flax grown at Saskatoon. The significant difference between the cultivars for this component disappeared when phytic acid was expressed as percent of P. The location effect on some of the meal components, such as ash, starch (present in negligible concentration), and total minerals, though statistically significant, may be of little significance as these are not taken into consideration for feeding of linseed meal to livestock, although individual components of the mineral matter may be of some importance in human nutrition. The other significant location effect was on protein, total carbohydrates, acid-detergent fiber and phytic acid. The mean protein content of the three flax cultivars grown at Saskatoon was 44.6%, as compared to 43.6% grown at Morden. Similarly, the total carbohydrate content of flax grown at Saskatoon was higher than that of flax grown at Morden (32.0% vs 29.2%). In contrast, flax grown at Morden had higher acid-detergent fiber (15.6% vs 14.8%) and phytic acid (3.0% vs 1.9%) than flax grown at Saskatoon. The location effect on linseed meal composition was probably largely influenced by climatic conditions.

TABLE 1

F Ratios (Obtained by Analysis of Variance) Showing Effects of Location, Cultivar and Location-Cultivar Interaction on Various Components of Laboratory-Prepared Samples of Linseed Meal

Component	Location	Cultivar	Location-Cultivar
Ash	2132.3^b	33.2^b	14.6^b
Protein	7.9^a	1.3	0.4
Total carbohydrates	21.3^b	2.3	0.5
Starch	128.1^b	4.5	6.5
Total dietary fiber	6.4	3.9	3.7
Acid detergent fiber	13.3^a	0.8	1.9
Cellulose	1.2	1.2	0.6
Lignin	0.7	0.1	4.6
Phytic acid	1496.3^b	13.0^a	8.3^a
Phytic acid-phosphorous	0.1	1.8	2.4
Pentosans	0.9	1.6	6.0^a
Total amino acids	5.7	2.7	0.4
Total minerals	153.0^b	4.6	0.2

^{a}P = 0.05. ^{b}P = 0.01.

Table 2 shows the mean, standard deviation and coefficient of variability (CV) of various components of LM and CM. The proximate composition of the two types of meal may be considered first. The means for LM and CM were not statistically significant for ash, starch, total dietary fiber, cellulose, phytic acid (% and % of P) and pentosans, although the CV for these components varied considerably. Only the means for protein, total carbohydrates, acid-detergent fiber and lignin were statistically significant. CM were considerably lower in protein than LM. The protein content of linseed meal may vary considerably, and values as high as 50–53% have been reported previously for individual samples of linseed meal (1,8). The mean protein content of 19 Egyptian samples

TABLE 2

Mean (X), Standard Deviation (SD_X) and Coefficient of Variability (CV) of Various Components of Laboratory-Prepared and Commercially-Obtained Samples of Flax

Component	Laboratory prepared meal (LM) (n = 6)			Commercial meals (CM) (n = 4)		
	$\bar{X}^a$	SD	CV	$\bar{X}^a$	SD	CV
Proximate, %						
Ash	6.4	1.0	15.6	7.1	0.6	8.5
Protein	43.9[c]	1.1	2.5	37.5[c]	2.5	6.7
Total carbohydrates	30.6[b]	1.7	5.6	34.7[b]	2.6	7.5
Starch	0.4	0.1	25.0	2.2	1.8	81.8
Total dietary fiber	39.6	0.5	1.3	41.5	2.5	6.0
Acid detergent fiber	15.2[b]	0.6	3.9	18.3[b]	1.9	10.4
Cellulose	9.9	0.4	4.0	11.6	2.1	18.1
Lignin	5.7[b]	0.4	7.0	6.5[b]	0.6	9.2
Phytic acid	2.4	0.6	25.0	2.7	0.2	7.4
Phytic acid-phosphorous	69.1	1.5	2.2	70.8	3.5	4.9
Pentosans	8.9	0.6	6.7	8.7	0.6	6.9
Minerals, mg/g						
Sodium	0.6	0.2	33.3	0.7	0.3	42.9
Potassium	12.1	2.4	19.8	13.4	1.6	11.9
Calcium	4.5	0.6	13.3	5.1	1.5	29.4
Magnesium	6.1	0.5	8.2	6.4	0.3	4.7
Phosphorus	9.9	2.6	26.3	10.7	1.3	12.1
Sulphur	4.0	0.1	2.5	4.1	0.2	4.9
Minerals, μg/g)						
Zinc	123.2	30.7	24.9	103.7	24.7	23.8
Iron	207.6	29.0	14.0	212.4	48.1	22.6
Copper	20.0	2.5	12.5	22.2	2.5	11.3
Manganese	58.5	9.7	16.6	54.0	2.1	3.9
Total	37.6	6.5	—	40.8	5.3	—
Monosaccharides, %						
Arabinose	4.2	0.4	9.5	4.3	0.4	9.3
Fucose	0.4	0.1	25.0	0.4	0.1	25.0
Galactose	4.5	0.4	8.9	4.6	0.8	17.4
Glucose	12.2	0.6	4.9	13.2	1.3	9.8
Rhamnose	1.1	0.2	18.2	0.9	0.1	11.1
Xylose	6.1	0.6	9.8	6.9	0.9	13.0
Total	28.5	2.3	—	30.3	3.6	—
Amino acids, g/16 g N						
Alanine	5.4	0.2	3.7	5.5	0.4	7.3
Arginine	11.8	0.6	5.1	11.1	0.6	5.4
Aspartic acid	12.5	0.5	4.0	12.4	0.7	5.7
Cystine	3.8	0.3	7.9	4.3	0.3	7.0
Glutamic acid	26.3	1.0	3.8	26.4	1.4	5.3
Glycine	7.0	0.3	4.3	7.1	0.4	5.6
Histidine	2.9	0.2	6.9	3.1	0.2	6.5
Isoleucine	5.2	0.2	3.9	5.0	0.4	8.0
Leucine	6.8	0.3	4.4	7.1	0.5	7.0
Lysine	4.1	0.1	2.4	4.3	0.6	14.0
Methionine	2.2	0.1	4.5	2.5	0.2	8.0
Phenylalanine	5.3	0.2	3.8	5.3	0.3	5.7
Proline	5.2	0.4	7.7	5.5	0.4	7.3
Serine	5.8	0.2	3.4	5.9	0.4	6.8
Threonine	4.9	0.2	4.1	5.1	0.4	7.8
Tryptophan	1.8	0.1	5.6	1.7	0.1	5.9
Tyrosine	2.9	0.1	3.5	3.1	0.2	6.5
Valine	5.6	0.2	3.6	5.6	0.4	7.1
Total	119.5	5.2	—	121.0	7.9	—
Vitamins, IU/100 g						
A	18.8			10.7		
E	0.6			0.5		
Vitamins, mg/100 g)						
B1	0.5			0.2		
B2	0.2			0.2		
B3	9.1			7.6		
B6	0.8			0.6		
B12	0.5			0.4		

[a] Significant difference between the two means calculated by a t-test. [b] $P = 0.05$. [c] $P = 0.01$.

of linseed meal was 32.8%, calculated on moisture and oil-free basis (7). CM contained more total carboyhydrates than LM. The data confirmed that linseed meal, like other oilseed meals, contained less than 1% starch, the range in the six LM was 0.2-0.5%. For some unknown reason (probably contamination), two of the four CM contained 3.4 to 3.9% starch. Such a range was responsible for the extremely large CV of these samples. Values for starch in linseed meal have not been reported in the literature, and erroneously included in the nitrogen-free extract (6-9). The two types of meal contained 30.6-34.7% total carbohydrates (significantly different), which was in the range for the nitrogen-free extract reported by the above workers.

CM were more fibrous than LM, and as a result contained more acid-detergent fiber (18.3 vs 15.2%) and cellulose (11.6% vs 9.9%). Linseed meal contains about 30% neutral detergent fiber (D. A. Christensen, personal communication). LM and CM contained similar levels of total dietary fiber (39.6-41.5%), which was much higher than present in some of the cereal products analyzed by the same procedure as used in the present study (27), or by an alternate procedure (28). Obviously, the high total dietary fiber content of linseed meal was largely due to its mucilage content.

LM were analyzed for six major (Na^+, K^+, Ca^{2+}, Mg^{2+}, P and S) and four minor (Zn^{2+}, Fe^{+3}, Cu^{2+}, and Mn^{+7}) elements (Table 2). The ranges for the elements in LM and CM were generally similar, and none of the means were statistically significant. Stitt (10) reported that 100 g of flax provided 100% of the US recommended daily allowance (RDA) of K^+ and Mn^{+7}, 87% of Mg^{2+}, 57-65% of Fe^{+3} and P, and 13-35% of Zn^+, Ca^{2+} and Cu^{2+}. Linseed meal is thus particularly deficient in Zn^{2+}, Ca^{2+} and Cu^{2+}. Although the mineral composition of different products is difficult to compare, the data suggest that LM contained several-fold more K^+, Ca^{2+}, Mg^{2+} and P than does wheat (29). The concentrations of Zn^{2+}, Fe^{+3}, Cu^{2+} and Mn^{+7} were 1- to 4-fold greater in LM than in wheat.

LM and CM had similar monosaccharide composition, the largest CV was obtained for fucose, which was the minor sugar. Glucose (may be slightly contaminated with fructose) was the major sugar, followed by xylose, galactose, arabinose, rhamnose and fucose. The sum of arabinose and xylose multiplied by 0.88 indicated the pentosan content of the meals (30). These values were 9.1 and 9.9% for LM and CM, respectively, and were similar to the mean pentosan contents (8.7-8.9%) obtained colorimetrically. The total sugar content of the meals (28.5-30.3%) was only slightly lower than the total carbohydrate content (30.6-34.7%), and with starch formed the nitrogen-free extract reported by other workers (4,6-9).

The amino acid composition of the LM and CM was nearly identical (Table 2). The largest CV were obtained for cystine and proline in LM and for lysine in CM. The meals were low in lysine and methionine compared to the WHO (31) requirement. Thus linseed meal needs to be supplemented with these amino acids for monogastric feeds. The amino acid composition of LM and CM was comparable to amino acid data of individual samples of linseed meal reported previously (8,32).

One sample each of LM (NorMan) and CM (Alberta Linseed, Medicine Hat) was analyzed for vitamins A, the B complex and E. The major difference between the two meals appeared in vitamin A. However, because of the single determination, the precision of the method was not known. Vitamin composition of linseed meal has not been generally reported. Linseed meal may be deficient in some of the components of the B complex (10).

Flax hull. In flax, the true hull or spermoderm is covered on the outside by the epiderm, containing the mucilage, and on the inside by the endosperm. The true hull is difficult to separate and may get contaminated with endosperm and the cotyledons. Nevertheless, with epiderm and endosperm, the hull may constitute about one-third of the seed (4). This value may vary a great deal depending upon the cultivar and the method of hull separation, and not correspond to the anatomical fractions of flax seed. A more recent study (33) reported 38-39% hull and 50% cotyledons in manually dissected flax seed. In the present study, the two stage liquid cyclone process described by Sosulski and Zadernowski (24) for the separation of hull and flour from canola gave 37.5% hull and 62.4% flour in flax (the values were adjusted to 100% oil-free and moisture-free meal). The corresponding values for hull and flour in canola and soybean were 18.6 and 81.3%, and 16.1 and 83.8%, respectively (Table 3). The hull percentage obtained for canola was about one-half of that reported earlier (24). To confirm that the difference was not due to the process, canola seeds were manually dehulled, and the hulls and the cotyledons expressed as percent of the total seed weight on "as is" basis. The values obtained were 13 and 87% for hull and cotyledons, respectively, which were closer to those given for canola in Table 3 than obtained by Sosulski and Zadernowski (24). The liquid cyclone process thus gave a reasonable separation of hull and flour fractions in the three oilseed species.

Table 3 gives the comparative composition of flax, canola and soybean hulls. In each case, the hull contained less than 1% oil, about 20% protein, which was probably partly due to contamination by endosperm and even the cotyledons. The protein content of flax hull was identical to that reported earlier (4).

TABLE 3

Yield and Composition of Flax, Canola and Soybean Hulls Obtained by a Liquid Cyclone Process

Yield/composition	Flax	Canola	Soybean
Yield, %			
Hull	37.5	18.6	16.1
Flour	62.4	81.3	83.8
Total	99.9	99.9	99.9
Composition, % dry basis			
Oil	0.9	0.2	0.4
Protein	20.3	19.9	20.3
Arabinose	5.0	5.7	4.6
Fucose	0.8	0.3	0.2
Galactose	5.1	2.7	5.3
Glucose	11.4	8.3	19.5
Rhamnose	1.5	0.4	0.5
Xylose	9.1	1.2	4.6
Pentosans	12.4	6.1	8.1
Galacturonic acid	11.1	—	—

Flax hull, like canola and soybean hulls, contained six monosaccharides, the major sugar was glucose and the minor fucose in each case. Xylose was the next major sugar of flax hull and with arabinose formed 43% of the total sugars, compared to 37% in canola and 26% in soybean. The arabinose content of the three hulls was not that different, but flax hull contained about two and eight times more xylose than soybean and canola hulls, respectively. As arabinose and xylose are major components of pentosans, flax hull contained 1.5- to 2-fold more pentosans than did canola and soybean hulls, calculated as the sum of arabinose and xylose multiplied by 0.88 (30). Flax hull may contain 2–7%, by weight of dry seed, mucilage present in the outer endosperm (4). The structure of linseed mucilage, a mixture of branched chain polysaccharides, has been described previously (34,35).

TABLE 4

Water-Hydration Capacity of Flax, Canola and Soybean Meal, Flour and Hull Fractions

Species	Water-holding capacity of each fraction[a]		
	Meal	Flour	Hull
Flax	7.9	2.0	13.0
Canola	2.1	1.7	1.9
Soybean	1.8	2.2	2.7

[a]Values are mg/g on oil-free and moisture-free basis.

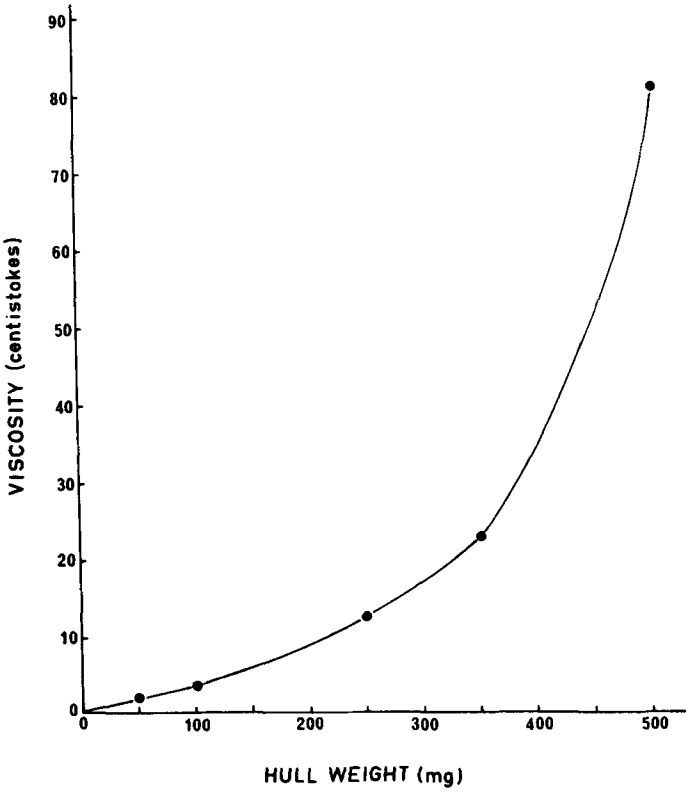

FIG. 1. The relationship between extract viscosity and hull concentration in flax. Different weights of hull were extracted with 15 ml of water at ratios varying from 1:30 to 1:300.

It consists of an acidic and a neutral component, the former containing L-galactose, L-rhamnose, L-fucose and D-galacturonic acid, and the latter mainly (1→4) linked D-xylose units (35). All of these sugars and galacturonic acid were identified in flax hulls (Table 3).

The linseed meal was highly hygroscopic, as shown by its water-hydration capacity (Table 4). The flax meal absorbed 8-fold and the hull 13-fold their weights of water. The water-hydroation capacity of flax flour was only two-fold, and similar to those of the meal, flour and hull fractions of canola and soybean. The present data confirmed earlier studies that the water-holding capacity of linseed meal was essentially due to mucilage present in the hull.

The flax hull mucilage was more soluble in water than in 0.1 M Tris-HCl buffer, pH 8.6 or in acidic buffer (0.1 M HCl-KCl, pH 1.5), as shown by its extract viscosity. The viscosity values for the three solvents were 13, 10 and 5 centiStokes, respectively. The extract viscosity was stable for at least 30 min at 25°C, suggesting no autolysis or enzymatic degradation of the mucilage. The relationship between hull concentration and extract viscosity was curvilinear (Fig. 1).

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