Composition of Several Types of Safflower Seed

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Abstract

The residue remaining after commercial extraction of oil from safflower seed has a greater potential as a source of animal feed or human diet supplement than is presently being realized. Safflower seed hull, kernel, and meal were analyzed to provide more information regarding their nutritive possibilities. Commercial and experimental normal hull varieties and experimental thin hull and striped hull varieties were hand separated into hull and kernel fractions and both fractions analyzed for protein, fat, fiber, ash, and amino acids. Samples of partially decorticated commercial meal and undecorticated meal, hulls, and defatted kernel from striped hull seeds were analyzed for protein, fat, fiber, ash, lignin, pentosans, anhydrouronic acid, total and reducing sugars, and amino acids. Cellulose was calculated by difference. A new factor for converting nitrogen to protein for summative analyses of safflower seed was calculated. These analyses indicate that about 15% of the nonfiber, nonash, nonprotein part of the defatted safflower kernel is of unknown composition.

Introduction

SAFFLOWER (*Carthamus tinctorius L.*), a relatively new crop in the United States, having increased to over 300,000-ton production from 8,000 tons in 1949 (1) has been grown almost entirely for its oil content.

One-half to two-thirds of the weight of the seed (achene) after removal of the oil has brought in less than 15% of the processors' gross return (2). Although the oil- and hull-free kernel contains from 58–72% crude protein (N × 6.25), commercially it is usually mixed with a large amount of the high fiber, low energy hull (pericarp and seed coat). New varieties are being developed with different fatty acid compositions and with different hull types, which have much lower hull percentages. Newer techniques for decorticating the seed before extracting the oil and for separating the hull fragments from the expressed kernel are also being developed to upgrade the meal. As the fiber content of the extracted meal is lowered, it becomes more valuable as a poultry feed and even becomes a potential high protein human food supplement (3). To utilize the protein and meal to the best advantage, more quantitative data are needed on the nutritionally and economically important components of the various safflower seed types.

In the present work, safflower seeds have been hand dissected and the hulls and kernels analyzed separately for nitrogen, crude fiber, ash, and ether extractables. Samples of commercially defatted meal and laboratory prepared hulls and kernels have been analyzed for the above plus lignin, pentosans, total and reducing sugars, anhydrouronic acid, and amino acids. Values for WRRL[3] Cellulose were calculated. Some preliminary information on the free sugars of the kernel are reported.

Materials and Methods

Seed composition was studied on 24 seed samples of commercial and experimental lines and varieties. Twelve samples were from normal hull varieties and lines, including 7 samples from commercial varieties and 5 samples from experimental lines having special hull and oil characteristics due to mutant genes. The other 12 samples were from experimental varieties and lines having a lower hull percentage due to mutant hull genes, namely the thin hull, striped hull and pigmentless, striped hull (4,5).

Small samples of seed were split by an apparatus modeled after a safflower seed cutter developed at the University of Arizona (6). It consists of a pair of hinged hardwood blocks. The lower has safflower seed-sized depressions in rows, the depressions in each row being connected and bisected by a narrow slot. The upper block has razor blades set into it in such a manner that they mesh with the slots in the lower block. Bringing the two blocks together splits the seeds which are then hand separated into hull and kernel.

A large sample of clean hull and another of clean kernel were prepared from brown striped hull seed by the following procedure. The sample of kernel was prepared by first cracking the seeds in a modified Waring blendor and then floating off the hulls on an air column. Two of the blendor blades were removed entirely; the other 2 were turned so that they acted as paddles rather than as cutters. The speed at which the blendor was operated was determined by the type of hull being cracked. Hulls were removed from small (25 ml or less) batches of the cracked seed in a 3 × 100 cm glass column with a goose neck at the top and a fine mesh wire screen basket trap at the end. Air was introduced tangentially at the bottom and the velocity increased until the hulls were carried over and caught in the trap. The heavy material remaining behind was a mixture of broken kernels and some small bits of hull. The larger pieces of kernel were separated by screening, leaving only small pieces of kernel mixed with small pieces of hull. Careful air classification of this mixture made possible the preparation of a large sample containing the kernel and including only a very small amount of hull.

Samples from 6 defatted meals were analyzed. Five were defatted meals from normal hull safflower that had been partially decorticated commercially, and 1 was an undecorticated but defatted experimental meal from brown, striped hull seed.

Analytical Methods

Total Solids. 16 hr at 70C in vacuum.

Nitrogen. Kjeldahl method (7).

Protein. Nitrogen × 6.25 (7) or 5.45.

Ether Extractables. 16 hr extraction with ethyl ether in a reflux type extractor (7).

Crude Fiber. Digestion of ether extracted residue

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with 0.255 N H_2SO_4 for 30 min followed by digestion with 0.313N NaOH for 30 min (7).

Ash. 600C for 1 hr.

Lignin. The Ellis-Matrone-Maynard method (8) with slight modifications as follows: Extraction thimbles with fritted disks were used instead of Gooch alundum crucibles. The hydrolyses in 5% and 3% sulfuric acid were carried out in an autoclave for 10 min at 20 lb steam (9) rather than refluxing for an hour. Ashing was done at 500C for 2 hr.

Pentosans. The method of Adams and Castagne (10) was used. Colors were measured in an Evelyn colorimeter with a 515 millimicron filter. Furfural estimated from a standard curve times 1.55 (11) equals xylose.

Anhydrouronic Acid (AUA). The samples were extracted 1 hr with 80% ethanol at boiling point and overnight at room temperature. The residue was extracted twice with 0.5% ammonium oxalate solution at 85C, once for 4 hr and again overnight. The combined ammonium oxalate extracts were acidified by passing them through a Dowex 50 (H^+) cation exchange column and then were concentrated to 50 ml in a vacuum rotary concentrator at 60C. Five volumes of 95% ethanol added to the concentrate precipitated the pectin. After centrifuging, washing with 95% ethanol, and drying the precipitate, uronic anhydride was determined on it by the carbazole method of McComb and McCready (12).

Sugars. Sugars were extracted by 80% ethanol, the alcohol removed by evaporation and the aqueous solution treated with ion exchange resins. The sucrose was hydrolized by invertase and the reducing sugars determined by the Shaffer-Somogi method (7).

Amino Acids. These were determined by the ion exchange column chromatography method of Kohler and Palter (13) using a Phoenix Model K-8000 amino acid analyzer. Results obtained by Lyman, et al. (14) are included in Table II for comparison.

A factor of 6.25 has been used by generations of nutritionists for converting nitrogen determined by analysis to crude protein. This factor is accurate in an absolute sense only for a protein or protein mixture which contains exactly 16% nitrogen. Although practically all proteins have other than 16% nitrogen, the use of this factor for comparative purposes is very useful to nutritionists and the feed industry, and undoubtedly will continue to be used for many more years. However, for summative analyses such as we use for calculating WRRL Cellulose, a factor is required to give us the true amount of protein. The nitrogen content of the 19 amino acid residues (e.g. protein) was calculated for the 13 samples of kernel (Table II) and the average was found to be 18.35%. Nitrogen recovery averaged 94%. The reciprocal of 18.35 multiplied by 100 is the protein factor, which was found to be 5.45 ± 0.020. All of the amino acid was considered to be protein amino acid for this calculation. The protein factor for the safflower hull sample was found to be 5.48. Since the hull contributes only 10% of the total protein of the seed the factor 5.45 is applicable to whole or partly decorticated meals as well as the kernel.

Doubly Extracted Residue. The method of Henderson (15) as modified by Binger et al. (11) was used. Soxhlet extraction with benzene-alcohol was followed by a 4 hr and a 16 hr, 85 C, 0.5% ammonium oxalate solution extraction. Air dry samples and 95% ethanol were used for the Soxhlet extraction. Centrifugation was used in place of all but the last filtration.

WRRL Cellulose (16). All of the major plant constituents but 1 are satisfactorily accounted for by direct analytical procedures. The methods commonly used for the determination of cellulose in forage materials all have shortcomings (17). Either lignin or polyuronide materials are not completely eliminated, or only a part or none of the cellusans are included. These latter closely related intermediate chain length, hot water insoluble carbohydrate polymers are probably nutritionally similar to α-cellulose and so would best be included with it.

TABLE I

Safflower Seed Composition[a]

	Crude fat %	Crude protein (N × 6.25) %	Crude fiber %	Ash %	NFE %	Kernel[c] %
Commercial varieties						
Gila (4)[b]						
Whole seed	38.1	16.7	22.3	2.6	20.3	62.0
Hull	3.2	4.3	57.1	2.0	33.4	.....
Kernel	60.9	24.9	1.6	3.1	9.5	.....
Oil free whole seed	.....	27.0	35.9	4.2	32.9	.....
Oil free kernel	.....	64.0	4.1	7.9	24.0	.....
U-5 (1)						
Whole seed	38.5	17.2	21.1	2.3	20.9	63.9
Hull	2.2	5.0	58.4	1.4	33.0	.....
Kernel	61.8	25.4	1.5	2.9	8.4	.....
Oil free whole seed	.....	28.0	34.3	3.7	34.0	.....
Oil free kernel	.....	66.4	3.9	7.5	22.2	.....
US-10 (1)						
Whole seed	36.8	19.4	22.3	2.5	19.0	63.3
Hull	1.4	3.8	60.0	1.6	33.2	.....
Kernel	59.0	29.4	1.5	3.2	6.9	.....
Oil free whole seed	.....	30.7	35.3	4.0	30.0	.....
Oil free kernel	.....	71.7	3.7	7.7	16.9	.....
Frio (1)						
Whole seed	40.1	15.4	20.8	2.3	21.4	64.6
Hull	2.7	4.1	60.4	2.2	30.6	.....
Kernel	64.0	23.0	1.0	2.6	9.4	.....
Oil free whole seed	.....	25.7	34.7	3.8	35.8	.....
Oil free kernel	.....	65.6	2.8	7.3	24.3	.....
Experimental varieties						
Normal hull Hi-stearic (1)						
Whole seed	27.8	20.0	31.8	2.7	17.7	52.3
Hull	1.6	3.1	65.8	0.9	28.6	.....
Kernel	52.0	35.7	1.1	4.4	6.8	.....
Oil free whole seed	.....	27.7	44.0	3.7	24.6	.....
Oil free kernel	.....	74.4	2.4	9.2	14.0	.....
Normal hull Hi-oleic (1)						
Whole seed	22.6	17.2	35.5	2.3	22.4	43.9
Hull	2.5	5.2	63.9	0.9	27.5	.....
Kernel	50.8	33.9	2.1	4.3	8.9	.....
Oil free whole seed	.....	22.2	45.8	3.0	29.0	.....
Oil free kernel	.....	69.0	4.2	8.8	18.0	.....
Normal hull equal oleic-linoleic (1)						
Whole seed	27.3	17.9	30.6	2.4	21.8	50.1
Hull	2.4	3.9	62.1	1.0	30.6	.....
Kernel	53.9	33.2	1.3	3.9	7.7	.....
Oil free whole seed	.....	24.6	42.1	3.3	30.0	.....
Oil free kernel	.....	71.9	2.9	8.5	16.7	.....
Other normal hull mutantvarieties (2)						
Whole seed	37.8	17.3	21.5	0.7	22.7	63.6
Hull	5.0	5.5	55.4	2.2	31.9	.....
Kernel	58.1	24.7	2.8	3.1	11.3	.....
Oil free whole seed	.....	27.7	34.5	4.3	33.5	.....
Oil free kernel	.....	58.3	6.6	6.8	28.3	.....
Pigmentless striped hull (1)						
Whole seed	42.8	22.5	13.6	3.5	17.6	74.7
Hull	5.6	8.6	46.2	5.1	34.5	.....
Kernel	55.9	27.4	2.7	3.1	10.9	.....
Oil free whole seed	.....	39.3	23.8	6.1	30.8	.....
Oil free kernel	.....	62.2	6.0	6.9	24.9	.....
Brown striped hull (8)						
Whole seed	47.7	20.3	11.7	3.4	16.9	75.9
Hull	5.7	8.4	46.9	4.9	34.1	.....
Kernel	62.7	24.8	0.9	3.1	8.5	.....
Oil free whole seed	.....	38.6	22.4	6.6	32.4	.....
Oil free kernel	.....	66.4	2.4	8.3	22.9	.....
Thin hull (3)						
Whole seed	47.2	21.1	11.2	3.3	17.3	76.0
Hull	5.1	10.0	45.3	5.1	34.5	.....
Kernel	62.6	25.5	0.9	3.0	8.0	.....
Oil free whole seed	.....	40.0	21.2	6.4	32.4	.....
Oil free kernel	.....	67.8	2.3	7.9	22.0	.....

[a] All results on a moisture free basis.
[b] Number of samples represented.
[c] Hull plus kernel equals 100%. The loss (average 3.0%) was assumed to be of the same ratio of hull to kernel as the rest of the sample.

TABLE II

Amino Acids of Safflower Seed, gm/16 gm Nitrogen

	Protein % (N × 6.25)[a]	Lysine	Histidine	Ammonia	Arginine	Aspartic acid	Threonine	Serine	Glutamic acid	Proline	Glycine	Alanine	Cystine	Valine	Methionine	Isoleucine	Leucine	Tyrosine	Phenylalanine	Tryptophan	Protein Factor
Defatted, hand separated kernels																					
Normal hull seed																					
Commercial varieties																					
Gila (3)[b]	66.4	2.72	2.34	2.42	9.52	9.33	2.96	4.24	20.55	3.80	5.46	4.03	1.86	5.44	1.59	3.84	5.97	2.91	4.20	1.15	5.46
U-5 (1)	66.4	2.83	2.43	2.39	9.74	9.40	3.17	4.38	19.69	3.56	5.38	4.06	1.80	5.43	1.75	3.83	6.13	3.03	4.21	0.93	5.44
US-10 (1)	71.7	2.63	2.28	2.42	9.69	9.47	2.84	4.16	19.97	3.87	5.09	3.90	1.62	5.24	1.28	3.53	5.87	2.86	4.21	0.90	5.42
Frio (1)	65.6	2.83	2.44	2.40	9.32	9.24	3.04	4.19	19.49	3.78	5.43	4.03	. ....	5.49	.....	3.86	5.98	2.91	4.15	.....	.....
Experimental varieties																					
Normal hull Histearic (1)	74.4	2.53	2.50	2.59	9.76	9.23	2.57	3.72	20.14	3.52	4.92	3.67	.....	5.45	.....	3.76	5.85	2.78	3.96	.....	.....
Normal hull Hi-oleic (1)	69.0	2.83	2.42	2.42	9.66	9.17	2.86	4.01	19.82	3.58	5.11	3.81	.....	5.25	.....	3.70	5.79	2.74	4.06	.....	.....
Normal hull equal oleic-linoleic (1)	71.9	2.61	2.42	2.48	10.09	9.44	2.92	4.37	21.24	3.92	5.19	3.95	.....	5.36	.....	3.76	6.12	3.03	4.16	.... .	.....
Other normal hull mutants (2)	58.3	2.91	2.60	2.59	10.32	10.29	3.24	4.74	22.39	3.83	5.48	4.51	2.01	5.96	1.69	4.15	6.57	3.16	4.53	1.08	5.47
Seeds with low hull content																					
Pigmentless, striped hull (1)	62.2	2.68	2.51	2.61	10.19	10.25	3.06	4.72	20.45	3.74	5.65	4.39	2.04	5.81	1.61	4.02	6.51	3.09	4.50	1.15	5.42
Brown striped hull (5)	65.9	2.73	2.41	2.46	9.67	9.69	2.96	4.34	20.50	3.88	5.47	4.10	1.81	5.47	1.61	3.89	6.05	3.00	4.90	1.08	5.45
Thin hull (3)	67.8	2.75	2.39	2.45	9.58	9.40	3.01	4.27	20.12	3.84	5.36	4.06	. ....	5.46	.....	3.88	6.07	3.04	4.29	.....	.....
Hulls																					
Gila (1)	4.0	2.86	1.22	2.50	2.87	6.38	3.11	4.36	7.82	3.37	4.53	3.29	.....	4.34	.....	3.14	4.62	1.16	3.25	.....	. ....
Brown striped hull	8.1	3.21	1.58	2.15	3.43	7.56	3.39	4.75	8.80	3.78	5.01	3.79	1.65	4.79	1.04	3.47	5.09	1.46	3.70	0.43	5.48
Thin hull (1)	10.2	3.07	1.33	2.30	3.09	7.30	3.03	4.48	7.78	3.26	4.46	3.32	.....	4.31	.....	3.15	4.58	1.52	3.47	.....	.....
Safflower meal																					
Commercial partially decorticated, normal (5)	48.0	2.84	2.32	2.42	8.69	9.13	3.10	4.36	19.34	3.93	5.46	4.17	1.70	5.46	1.62	3.97	6.13	2.48	4.32	.....	5.45
Experimental undecorticated, brown, striped hull (1)	38.1	2.63	2.23	2.56	8.34	9.22	2.93	4.17	18.56	3.68	5.27	3.97	1.63	5.32	1.38	3.80	5.86	2.39	4.29	.....	5.41
Results reported by																					
Lyman, et al.[c]	22.1	2.71	1.99		7.78		2.94							4.93	1.54	3.85	5.52		5.25	1.18	.....

[a] Moisture and oil free basis.
[b] Number of sample represented.
[c] (14)

TABLE III

Analyses of Commercial Safflower Seed Cakes and Laboratory Prepared Hull and Kernel

	Protein N × 6.25	Protein N × 5.42	Fiber	Lignin	Pentosans	AUA	Ash	Fat	Total sugars	Reducing sugars	NFE[a]	WRRL cellulose[b]
Commercially defatted and partially decorticated meal from thick hull seeds (3)	48.5	42.1	15.4	7.6	9.5	1.1	7.8	1.5	4.65	0.33	26.8	14.7
Undecorticated defatted meal from brown, striped hull seeds (1)	39.3	34.1	25.1	11.6	10.4	2.5	6.6	1.5	3.28	0.26	27.5	18.9
Clean hull from brown, striped hull seed (1)	8.1	7.0	44.2	21.5	18.5	2.5	4.5	11.5	1.13	0.44	31.7	32.1
Laboratory defatted clean kernel from thin hull seed (1)	58.8	51.0	5.7	3.2	4.6	1.1	9.9	1.0	7.61	0.44	24.6	6.1

[a] Calculated from Protein = N × 6.25.
[b] Calculated from Protein = N × 5.42.

The residue remaining after the mild treatment conditions of the dilute ammonium oxalate extractions is free of nearly all of the ether soluble materials, all of the hot water solubles, and the pectin. It includes a trace of ether soluble material, some protein, the hot water insoluble inorganic substances, and the nonuronide constituents of the cell wall, including pentosan, lignin, and cellulose. Therefore, if the ether solubles, protein (N × 5.45), ash, pentosans, and lignin are determined and their quantities subtracted from the amount of doubly extracted residue, the remainder should be the cellulose including the cellusans. We have designated this calculated cellulose value "WRRL Cellulose" to differentiate it from cellulose by direct analysis.

Results and Discussion

Analytical results for the 24 samples are summarized in Table I. The results from the various samples were grouped according to variety or to special oil or hull characteristic. Because several of the varieties and lines were grown at different locations and in different years, the values obtained may not be valid for comparing varieties and lines. However, the results do represent a good sampling of the extent of variability that exists among varieties and lines. The 3 samples of normal hull seed selected for their special fatty acid contents are plant introductions from Israel, Iran, and India. All have lower total oil content than the commercial normal hull seeds, both because of a lower kernel-to-hull ratio and because of a lower kernel oil content. Thin hull and striped hull seeds, on the other hand, have a higher oil content than do normal hull seeds mostly because of their favorable kernel-to-hull ratio. The oil reported from the hulls is due mostly to unavoidable contamination from kernel. Although the protein content of the defatted hull free kernel is quite variable, the sum of the oil and the crude protein (N × 6.25) of the kernels varies only between 82.8% and 88.4% for the 24 samples tested, denoting a high inverse correlation between the oil and protein content of the kernel. Also, the amino acid distribution within the kernel proteein (Table II) is quite uniform throughout all the samples. The amino acid pattern of the hull is somewhat similar to that of the kernel except that there is less of most of the amino acids from hulls. This is probably a reflection of the low recoveries of total nitrogen as amino acids from the hull (ca 60%) as compared with the kernels (90–95%). However, there is only about half as much histidine and tyrosine and a third as much arginine, glutamic acid, and tryptophan as from kernel. The form in which the remaining 40% of the nitrogen is found in the hull is an interesting subject for further investigation.

Samples of commercial 42% protein safflower meal produced by partial decortication run up to nearly 50% crude protein (N × 6.25, dry basis) (Table III). Undecorticated meals from striped hull varieties have about 38% crude protein (N × 6.25) as compared to about 22% crude protein (N × 6.25) for undecorticated meals from the thick hulled types. Complete removal of hull brings the protein content of both types up to about the same level (58–70% crude protein, N × 6.25, dry basis). The higher protein of the hulls of thin hull and striped hull varieties (up to 10%) is of interest but this protein is probably poorly available nutritionally because of the more than 20% lignin also present.

The apparent crude fiber of the kernels shown in Table II is undoubtedly partly due to fine pieces of hull which are extremely difficult to separate even in hand dissected samples.

The ash content of the kernels of different types of seed show little variation. The ash content of the hull varies inversely with hull percentage. When the hull percentage is reduced, it is the outer layers of the pericarp which is reduced; this suggests that the ash may be in the inner layers of the pericarp or seed coat.

The material remaining after subtraction of fat, crude protein (N × 6.25), fiber and ash has been traditionally lumped as nitrogen free extract (NFE). NFE thus includes nonnitrogenous organic compounds not soluble in ether but soluble in boiling dilute acid or alkali. These include the free sugars, starch, gums, organic acids, and pectin as well as part of the structural carbohydrates and lignin.

None of the several safflower kernel samples tested with iodine–potassium iodide gave any indication of the presence of starch. Sugars were determined in commercial meals from both normal and striped hull lines and in clean hull and kernel from striped hull seeds. Total sugars averaged 6.3% of the defatted kernel including 0.38% reducing sugars. Paper

chromatography indicated the presence of sucrose and raffinose in approximately equal amounts, with very small amounts, if any, of other sugars.

When the protein (N × 5.45), lignin, pentosans, AUA, ash, fat, total sugars, and WRRL Cellulose are totaled there is still 10–13% of the meal samples, 1.3% of the hull, and 15.4% of the kernel unaccounted for. This missing fraction is probably made up of organic acids, non cellulosic, hot water soluble hexosans, phenolics, or other constituents which may each be present in small amounts. It is an important and interesting part of the defatted kernel and needs further investigation as to its identity and nutritional value.

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