traction of jojoba flakes as there are no significant differences in mass velocity and extraction efficiency obtained with the two solvents. However hexane is the preferred solvent because it is more readily available, is lower in cost, and has a lower boiling point, which facilitates its removal from the products.

Three experiments were conducted to determine the filtration-extraction characteristics of uncooked jojoba flakes. Results were erratic in that mass velocities varied from about 200 to 1,600 and extraction efficiencies from 96.5 to 97.8 (residual lipids 1.9 to 3.2%). Use of uncooked flakes is not recommended for commercial filtration extraction operations.

Summary

Data for the application of the versatile filtration-extraction process to jojoba seed on a bench-scale has been presented. Based on experience with other oilseeds, there should be good correlation between the bench-scale and its commercial application. Moisture contents of the material during cooking were optimum at 10 and 15%. Mass velocities in excess of 2,000 and extraction efficiencies of over 98% were obtained. These results are considered suitable for commercial application. Hexane is recommended over heptane as the extraction solvent. The use of uncooked flakes is not considered feasible for large-scale application.

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A Simple Method for Evaluation of Heat Treatment of Soybean Meal

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EXTRACTED SOYBEAN MEAL is heat-treated in order to destroy heat-labile, antinutritional factors which impair the nutritional value of the meal. Proper heat treatment is capable of destroying toxic or undesirable factors in soybean oil meal, such as trypsin inhibitor (1, 2, 3), hemagglutinin (4, 5), saponin, a goitrogenic substance (6), an anticoagulant factor, a diuretic principle, and lipoxidase. The heating must not be applied too long or at too high a temperature since serious damage may occur to the quality of the meal. Essential components may be destroyed with a concomitant decrease in the biological value (7, 8, 9, 10).

The most widely used *in vitro* methods for testing efficiency of soybean meal toasting include measurement of urease activity (11), destruction of trypsin inhibitor, solubility of protein (in water or mild alkali), fluorescence (12, 13), ability of meal to absorb dyes containing a phthalein group (14, 15, 16), availability and extent of destruction of amino acids during the heating process or decrease in free basic amino groups in soybean meal (17, 18, 19). A collaborative study of the urease method (20) showed that inadequately heated meals gave a positive test, but the method should be considered as having limited value for testing proper toasting and of no value in detecting overheating (21, 22). Evans and St. John (23) followed changes in solubility of soybean proteins during progressive increases in severity of moist heat treatment. Simon and Melnick (24) showed that reduction in protein solubility were generally correlated to biological improvement of soybeans during heating. Smith *et al.* (25) studied the effect of the age of soybeans, time and temperature of extraction, steam treatment, pH, and presence of wetting agent on the peptization of soybean meals. Balloun *et al.* (22) correlated the results of several *in vitro* laboratory tests with the nutritional value of soybean meals when fed to chicks. A determination of fluorescence was considered adequate for testing overheating of solvent-extracted meals but was of little value in detecting overheated expeller meals.

During the manufacture of soybean meal a quick control is essential, and only simple methods which enable an immediate decision to be made are of value. On the other hand, the information which can be expected from such methods is, of course, limited. No single physical or chemical (and probably not even biological) test can provide an evaluation significant in respect to all possible applications and uses to which the meal may be put. The value of the meal depends—in addition to severity and length of heat treatment—on the method of preparation of the soybean meal (solvent, hydraulic, or expeller) and

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time of its storage (26, 27); on the species to be fed and their age; on composition of the diet, etc. Carpenter (28) therefore concludes that there are no chances of devising an ideal chemical test for over-all evaluation of the nutritive value of vegetable protein.

In this paper the proposed method measures changes in protein solubility during heating of soybean meal by determination of the refractive index of dilute sodium hydroxide extracts of meals. The test seems attractive as a screening test because of simplicity and speed. The method was compared with several *in vitro* tests for estimating the extent of heating in soybean meal, *i.e.*, procedures of determining protein solubility, urease activity, dye absorption (16), and available lysine (18).

Preliminary experiments in these laboratories have shown that amylase activity (as assessed by change in the maximum hot paste viscosity of a 10% suspension of flour and soyflour, at 10% level), measured with the Brabender Amylograph (29), could not be used to follow heat treatment of soyflour. Inactivation of amylase during commercial manufacture of soymeal was advanced even before significant changes in urease activity or protein solubility were noted. Completely untreated soyflour, produced in the laboratory, showed considerable starch liquefying properties.

The proposed method shows high correlation with the usual dilute alkali-extract procedure for determining protein solubility. The inherent limitations of the protein solubility test as well as of any simple chemical test, as mentioned before, must be allowed for in interpretation of the results.

Materials and Methods

Thirteen samples of solvent-extracted soybean meal, from seven sources were used in this study. Samples 3, 4, 5, 6, 7, 8, and 10 were locally produced, commercial soybean meals obtained during the last part of 1958 or during 1957 (Sample No. 6). Sample 9 was an imported, commercial soybean meal, produced in 1957. Whereas Samples 7 and 10 were mildly heat-treated (with the purpose of desolventizing), Samples 3, 4, 5, 6, and 8 were additionally toasted with the purpose of improving the biological value of the meal for chicks.

Samples 1 and 2 were experimental meals (prepared on pilot-plant scale) treated by additional heating and the addition of live steam to Samples 7 and 10, respectively.

Samples 11 and 12 were prepared from soybeans pulverized in the laboratory. The samples were extracted with petroleum ether (b.p. 40–60°C.). Sample 11 was prepared with a minimum of heating while Sample 12 was heated for 2 hrs. at 130°C. prior to extraction.

Additionally, an unheated soyflour, prepared from partly dehulled beans (Sample 13), was heat-treated for various times at 15 lbs. of pressure in a laboratory electrical autoclave. As the heating and cooling of the meal was rather long, the figures given in Table III are of comparative value only.

The proposed method was subsequently used in assessing heat treatment of soybean meal in a commercial toasting installation. After injection of live steam the meal was heated for 60 min., and the original moisture content was reduced from 15.7–22.8% to a final water content of 8.2–9.0%. The temperature of the meal after removal from the toaster was 70–80°C. Ten samples were drawn and tested.

The determinations of moisture, ash, protein, oil, and crude fiber were made according to A.O.C.S. Methods (30). Soluble protein was determined by the method of Smith *et al.* (25). The meals described in Table I were ground on a laboratory mill to pass an 80-mesh sieve, and the determinations were made on this material.

TABLE I

Description and Composition of Soybean Meals

No.	Description	Year	Composition [a]			
			Ash	Protein (Nx6.25)	Oil	Crude fiber
			%	%	%	%
1	Experimental	1958	6.9	49.2	0.9	5.8
2	Experimental	1958	6.1	50.5	0.4	5.4
3	Commercial	1958	6.3	50.9	1.1	5.5
4	Commercial	1958	6.9	50.8	0.7	4.6
5	Commercial	1958	6.4	47.5	0.7	4.2
6	Commercial	1957	6.5	51.7	1.0	5.2
7	Commercial	1958	6.3	49.3	0.5	6.4
8	Commercial	1958	6.4	48.9	0.7	5.2
9	Commercial	1957	6.5	48.9	0.5	7.0
10	Commercial	1958	6.1	51.2	0.6	5.4
11	Experimental	1958	5.3	48.8	0.8	5.3
12	Experimental	1958	6.1	50.0	0.1	5.9
13	Commercial	1958	6.6	53.2	0.8	2.5

[a] All figures expressed on moisture-free basis.

The samples were tested by the following procedure. Five grams were suspended in a 100-ml. solution in a stoppered Erlenmeyer flask and shaken for 1 hr. in a laboratory shaker. The contents of the flask were centrifuged for 5 min. at 1,500 r.p.m. The refractive index of the supernatant solution was determined (at 25°C.). Since all refractive index readings lay between 1.3300 and 1.3400, only the figures in the third and fourth decimal places were recorded. These appear in Tables II, III, and IV. Various modifications of the proposed method were tried. The time of extraction was varied between 30 and 120 min., the meal was suspended in water, 0.02 N NaOH, 2.5% lauryl sulphonate solution, and various ratios of meal to solution were tried. The greatest and most reproducible differences in protein solubility were obtained on shaking a 5-g. sample with 100 ml. of either dilute sodium hydroxide or lauryl sulphonate for at least 1 hr. Data reported in Tables II, III, and IV are those obtained under the above-mentioned conditions, using sodium hydroxide.

Description and composition of the tested meals are given in Table I.

Results

The results of various tests made in order to assess the heating of the meal are given in Table II. The effect of heating by autoclaving the meal are sum-

TABLE II

Assessment of Heat Treatment by Various Chemical Tests

Sample	Urease activity (change in pH)	Soluble protein (% total protein)	Refractive index [a]	Dye absorption [b]	Available lysine [c]
1	0.2	29.9	56	4.1	5.0
2	1.6	76.2	81	3.6	5.0
3	0.2	38.5	61	4.0	4.7
4	0.3	13.6	51	4.4	5.0
5	0.1	12.0	50	4.4	4.8
6	1.2	58.7	71	3.8	5.1
7	1.6	52.5	67	3.7	5.1
8	below 0.1	42.3	65	4.2	5.4
9	below 0.1	44.5	64	4.2	5.1
10	1.3	73.8	81	3.7	5.7
11	2.0	84.8	83	3.1	5.6
12	0.2	.....	62	4.2	4.7
13	1.6	87.1	89	.....	.....

[a] Figures in third and fourth decimal place following 1.33.
[b] Mg. of dye absorbed by 1 g. of soyflour.
[c] G./16 g. of N.

TABLE III

Effect of Increased Time of Autoclaving on Protein Solubility of Soymeal

Sample No.	Refractive index [a]	Soluble protein (as % of total protein)
13	89	87.1
13A	57	21.4
13B	53	16.5
13C	50	9.6
13D	50	9.7
13E	47	6.6

[a] Figures in third and fourth decimal place following 1.33.

marized in Table III. The results of tests made on commercially toasted samples are given in Table IV.

Discussion

The results of various methods, reflecting differences in soybean meals caused by heat treatment, are shown in Table II. Information obtained from the processors indicated that these meals indeed had been subjected to a wide range in intensity and duration of heat treatment.

If the soluble protein percentage is taken as a reference for comparison of all other procedures, it will be noted that the wide range in these values, which reflects reasonably well the range of treatment qualitatively described by the processors, is not reflected by a similar sensitivity to differences in the urease determinations. The value of urease activity appears to be limited to indicating meals which are largely underprocessed. It is not useful for over-heated meals.

The simplified refractive index method for determining soluble protein proposed here is highly and positively correlated ($r = +0.92$) with the soluble nitrogen determination (Figure 1). Properly heated meals gave refractive index readings for dilute sodium hydroxide extracts between 55 and 65; higher figures are associated definitely with underheating, and the lower numbers indicate overheating. However protein solubility determinations seem to be of limited value in assessing the extent of overheating.

Properly heated soyflour is expected to show dye absorption values between 4.0 and 4.3 mg. per gram of meal; lower values are exhibited by underheated, higher values by overheated meals. These figures refer to fresh meals. On storage the dye absorption capacity of the meal decreased (up to 0.2 mg. per g. of meal during the first six-month period). The results obtained show a possibility of rough evaluation of heating by this simple and rapid method. Its greatest value seems to be in its ability to detect grossly overheated meals.

The same applies to available lysine measurements. This method suffers from several additional limitations. The test is rather lengthy, and at the present moment it seems that the procedure needs additional standardization of testing conditions in order to obtain more reproducible results as well as elimination of erratic determinations from interfering components of the soymeal.

TABLE IV

Effect of Toasting on Protein Solubility and Dye Absorption of Soymeal

Sample	Soluble protein (as % of total protein)	Refractive index [a]	Dye absorption [b]
14	46.3	66	3.7
15	59.7	70	3.6
16	62.2	72	3.6
17	.....	69	3.7
18	.....	70	3.4
19	49.1	68	3.9
20	40.2	63	4.0
21	15.9	53	4.1
22	56.6	74	3.6
23	39.2	64	3.9

[a] Figures in third and fourth decimal place following 1.33.
[b] Mg. of dye absorbed by 1 g. of soymeal.

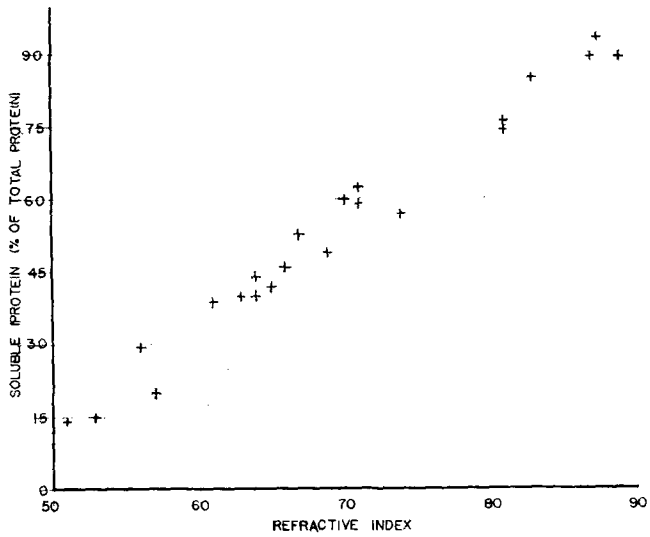

FIG. 1.

Summary

A simple and rapid method for estimating protein solubility of soybean meals on the basis of changes in the refractive index of dilute sodium hydroxide solution extracts was tested and found to be highly correlated with the usual protein solubility test. The method was compared with four *in vitro* tests of evaluating severity and adequacy of heating soybean meal. The value and inherent limitations of the various screening methods were compared.

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