Ethanol Extraction of Soybean Oil

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Abstract

Extraction of soybean flakes with 90, 95, 98 and 100% ethanol resulted in more rapid lipid and less rapid non-lipid removal with the increasing ethanol concentrations. There was little difference in the quality of the oil produced by the different solvents. Protein content of the residual meal averaged 52.1%.

Introduction

THE SOLVENT used in the commercial extraction of vegetable oils in the United States is a petroleum fraction boiling at about 150–160F and commonly referred to as "hexane." In Asiatic countries where imported petroleum solvents are expensive and where ethanol produced locally is available in adequate quantities there is considerable interest in the ethanol as a solvent.

Beckel and co-workers (4, 5) developed a continuous non-distillation method for extracting soybean oil by ethanol theoretically requiring only 0.7 of the energy required by the hexane method. Rao and Arnold (9, 11) have reported solubility data on 14 vegetable oils in aqueous ethanol. Extraction studies on both a glassware and pilot plant scale (10) indicate that cottonseeds may be satisfactorily extracted by ethanol.

Ethanol is said to produce a meal having a higher nutritive value and improved flavor (4). Rao (12) secured very good oil and meal by alcoholic extraction of prepressed peanut cake. Rao and Arnold (10) obtained a prime oil and a meal with very low gossypol content by extracting cottonseed with ethanol.

Ethanol extracts a considerable amount of non-lipid material but this is not necessarily a disadvantage. Mustakas et al. (8) report that they obtained improved bland products containing over 70% protein by washing defatted commercial soybean flakes with aqueous ethanols. The current study was initiated to secure data not previously available which would be needed in commercial ethanol extraction of soybean oil.

Experimental

Cracked soybeans containing 7–8% moisture were heated to 160F and rolled into flakes with an average thickness of 11 mils. The extractions were carried out in laboratory rate extraction apparatus similar to that used in previous studies in this laboratory (2), but with the extraction chamber 12 in. high by 2 in. in diameter allowing the use of 100g samples. Four concentrations of ethanol were used as solvents: absolute, 98% 95% and 90% alcohol. The extraction chamber and the incoming solvent were heated to 160F. The solvent passed through the flakes for 60 min at a rate producing 10 ml per minute of miscella. Samples were taken at 10 min intervals. The solvent was evaporated from each fraction under vacuum and the residue weighed. Since it was observed that a considerable amount of solids was being extracted by ethanol the residues were exhaustively extracted with ethyl ether, and the ether evaporated off to give the total lipids extracted. The residues remaining after extraction were dried, weighed and analyzed for the nitrogen content by the semi-micro Kjeldahl method. The results are shown in Table I.

TABLE I

Material Extracted from Soybeans by Ethanol (100g samples)

Extraction time, min	Lipids g	Non-lipids g	Nitrogen g × 6.25 [a]
100% Ethanol			
10	6.1171	0.3733	0
20	10.9072	0.7770	0.0174
30	14.4882	1.4311	0.0174
40	16.5361	2.0082	0.0578
50	17.9470	2.5583	0.0578
60	18.9845	3.0238	0.0652
98% Ethanol			
10	4.7844	0.8629	0.0224
20	8.7415	1.6404	0.0652
30	11.5084	2.3071	0.0845
40	13.7074	3.0245	0.1153
50	15.3106	3.6697	0.3153
60	16.4002	4.2887	0.4271
95% Ethanol			
10	3.1411	0.6017	0.0379
20	6.4115	1.4051	0.0917
30	9.1039	2.7542	0.1457
40	11.9103	3.3670	0.1708
50	14.0498	3.9234	0.2008
60	15.5742	4.4837	0.2288
90% Ethanol			
10	1.7711	0.9877	0
20	3.7631	1.8524	0.0579
30	5.4560	3.0434	0.0769
40	7.0351	4.2174	0.1250
50	8.5533	5.1925	0.1347
60	10.0336	5.9768	0.4167

[a] Protein equivalent.

Four batches of soybean flakes were extracted with the four ethanol solutions for two hr keeping other conditions same as heretofore indicated. The desolventized oil was filtered and the following determined: free fatty acids, color, saponification value and iodine values by the Official AOCS Methods (1) and neutral oil and phospholipid content by the method of Choudhury and Arnold (6). The protein content of the residual soybean meal was determined by the Kjeldahl method. Results are shown in Table II.

The amount of material extracted by each concentration of ethanol is given in terms of g of lipids, non-lipids, and nitrogenous material per 100g of soybean flakes in Table I. The sum of the lipids and non-lipids constitutes the total extractables. The nitrogen was determined on the non-lipid fraction. The data are cumulative.

The data show the increasing effectiveness of ethanol-water solutions in the extraction of both total extractables and lipids with an increase from 90–100% ethanol. These results are similar to those shown in previous studies with cottonseed (10) and peanuts

TABLE II

Quality of Extracted Soybean Oil and Residual Meal

Property	Ethanol concentration			
	100%	98%	95%	90%
Oil				
Free fatty acid, %	0.8	0.7	0.8	0.6
Color	7.5	8.5	8.0	8.2
Saponification value	191.8	192.2	191.5	190.8
Iodine value	135.0	135.5	134.8	135.1
Neutral oil, %	96.7	96.5	95.8	94.7
Phospholipid, %	3.3	3.5	4.2	5.3
Meal				
Protein, % [a]	50.9	51.1	54.0	52.5

[a] Air dried basis; original soybean flakes 43.3% protein (dry basis).

(7). The non-lipid extraction is in the reverse order being the greatest with the 90% ethanol perhaps in part because of the lower water solubility of both protein and carbohydrates in higher ethanol concentrations. The former may result from the denaturing effect of the ethanol such as was shown in previous studies in this laboratory on ethanol-extracted cottonseed (3).

The quality of the oil is shown in Table II. Except for the slightly higher phospholipid, and correspondingly lower neutral oil, content in the oils extracted by the 90 and 95% ethanol, the oils show no significant differences. The protein content of the different meals (Table II) is not significantly different but far below the over 70% value given by Mustakas et al. (8) for ethanol-washed hexane-extracted meal. Apparently when used as a solvent the ethanol preferentially dissolves the oil and cannot take up as much carbohydrate material.

Conclusions

In the extraction of soybean oil by aqueous ethanol lipids are removed more rapidly and non-lipids less rapidly as the concentration of the ethanol increases. There is little difference in the quality of the oil extracted by different concentrations of ethanol. The protein content of the extracted meal averaged 52.1%.

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• *Letter to the Editor*

Tetracyanoethylene Adduct of *Trans,Trans*-9,11-Octadecadienoic Acid

SEVERAL Diels-Alder adducts of *trans,trans*-9,11-octadecadienoic have been reported (1) from this laboratory. This acid reacts readily with nitroethylene, β-nitrostyrene, acrylic acid, acrylonitrile, acrolein, methacrolein, methyl vinyl ketone, methyl vinyl sulfone, and propargylic acid. We have now prepared the adduct with tetracyanoethylene (TCNE). This dienophile reacts readily with a wide variety of dienes (2). Reaction with *trans,trans*-9,11-octadecadienoic acid proved to be equally facile. The course of the addition could be followed by typical color changes (2) from the initial green of the tetrahydrofuran (THF) solution of TCNE through the dark red of the intermediate pi-complex to the final light yellow solution of the adduct. After recrystallization, the adduct was a slightly off-white crystalline solid, melting at 69.5–71C. The infrared spectrum was consistent with the Diels-Alder structure; absorption bands showed the presence of a carbocyclic ring, *cis*-carbon-carbon unsaturation, and nitrile and carboxylic acid groups.

Experimental

Trans,trans-9,11-octadecadienoic acid (4.2 g, 0.015 mole) and TCNE (1.9 g, 0.0148 mole) were each dissolved in 7.5 ml of THF. The acid was only partially soluble in this amount of solvent. The TCNE solution was dark green. After the two solutions were mixed, the mixture turned dark red and slowly became a light yellow (in about 5 min). The THF was evaporated under vacuum to leave a viscous gray-green residue. Ten milliliters of pentane-hexane was added and the mixture stirred until the adduct solidified to a white powder, which was filtered to give 5.8 g of solid. Recrystallization from benzene-pentane-hexane gave slightly off-white micalike crystals, mp 69.5–71C.

Anal Calcd. for $C_{24}H_{32}N_4O_2$: C, 70.55; H, 7.90; N, 13.72. Found: C, 70.43; H, 7.93; N, 13.60.

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