Technical

Solvent Extraction of Lipids from Soybeans with Acidic Hexane

T.P. HENSARLING and T.J. JACKS, Southern Regional Research Center,[1] P.O. Box 19687, New Orleans, LA 70179

ABSTRACT

Hexane and hexane containing 5% acetic acid (v/v) were used to extract lipids from soybean at room temperature and at 60 C. Hexane/acetic acid extractions yielded ca. 11% more total lipids and ca. 6–10% more neutral oil than did hexane extractions. Hexane/acetic acid extraction at room temperature yielded the same or slightly more neutral oil than did hexane at 60 C. Thirty-five times more phosphorus was extracted with hexane/acetic acid than with hexane; this phosphorus represented ca. 46% of the phospholipid phosphorus of soybean. Also, when hexane/acetic acid was used as the solvent, the separation of miscella from marc by filtration was about twice as rapid as the separation when hexane alone was the solvent.

INTRODUCTION

Storage oils of oil seeds are contained in intracellular packets called spherosomes (1,2). Physiochemical characterizations (2) and electron microscopical observations (3) proved that the limiting, half-unit membrane (4) of these organelles are acid labile. In a previous communication, we showed that this acid lability could be exploited to acheive thorough extraction of lipids from cottonseed (5). Addition of 2–25% (v/v) acetic acid to hexane resulted in the extraction of up to 9.8% more total lipids, up to 4.4% more neutral oil, and up to 10-fold more phosphorous than that extracted by hexane alone. Since soybeans are the most important cash crop in the USA, it was of interest to determine whether solvent extraction with acidic hexane offered similar advantages over hexane for lipid removal from soybeans as it did for cottonseed.

EXPERIMENTAL PROCEDURES

Extracting solvents were industrial hexane (Skellysolve B) and industrial hexane containing 5% (v/v) glacial acetic acid. Soybean meats (Forrest, 1978) were dry-milled in an Alpine American Contraplex (impact stud mill). The resultant comminuted meats were stored over silica gel in vacuo. Samples of the dried comminuted meats were extracted in fritted glass Buchner funnels of medium porosity (6 mL solvent/2 g meats), 5 min contact with constant stirring) and resultant miscellae were collected by filtration with reduced pressure for 2 min (6). In the cases where extractions were made at 60 C, the first extraction was made in a beaker in a mineral oil-in-sand heat sink. Subsequent extractions were made in the funnel using 60 C solvents. Each marc was extracted twice more with fresh solvent and the three miscellae from each sample were combined. Wt of the extracted lipids (total lipids) were determined after solvents were removed in vacuo.

Amounts of neutral oil in the miscellae were determined by methods reported previously (6,7).

Phosphorus was determined spectrophotometrically (8) after digestion of materials as described previously (6).

Filtration rates of miscellae through marcs were determined by extracting comminuted meats with solvent (18 mL solvent/6 g meats, 2 min contact with constant stirring) in fritted glass Buchner funnels with matched solvent void times, allowing to settle 5 sec, filtering with reduced pressure, and recording void time of miscellae from funnels.

RESULTS

Extraction of lipoidal materials from soybeans by hexane and hexane/acetic acid (5%, v/v) were compared. Extractions were performed at room temperature and at 60 C. Total lipids, neutral oil, and phosphorus contents of the miscellae were determined. Table I shows that acidic hexane, at room temperature and at 60 C, extracted significantly more total lipids (10.9% and 10.3%, respectively) and more neutral oil (9.5% and 6.0%, respectively) than did hexane alone at each temperature. Furthermore, acidic hexane at room temperature extracted an approximately equal amount of neutral lipid as did heated (60 C) hexane. Amounts of phosphorus extracted by acidic hexane were from 16- to 35-fold greater than amounts of phosphorus extraced by hexane alone (Table I).

Table II shows that the filtration rate of miscella through marc was approximately twice as fast when acidic hexane was used as the extraction solvent compared to hexane alone.

DISCUSSION

Beans were dehulled and comminuted at ambient moisture contents but were rendered bone-dry for extraction. This resulted in lowered phosphorus contents of the crude oils extracted by hexane (Table I). The phosphorus contents do not account for losses in purifying neutral oils from total lipids. For instance, apparent phospholipid content (P X 32) and "refining loss" (total lipid minus neutral oil) was 0.32 mg/g of meats and 23 mg/g of meats, respectively, for hexane extraction at room temperature and 10.34 mg/g of meats and 28 mg/g of meats, respectively, for acidic hexane at room temperature. Since free fatty acid amounts are constant, regardless of solvent acidity (5), then extraction of an additional, "strongly bound" neutral oil was accompanied by a disproportionate amount of phospholipid. This would happen if neutral and polar lipids of membranes were extracted. Indeed, earlier we found that

[1] ARS, USDA.

TABLE I

Components of Miscella[a]

Condition of extraction		Total lipids[b]		Neutral oil			
Temperature	% HOAc in hexane (v/v)	mg/g of meats	as % of lipids from 0% HOAc	mg/g of meats	as % of oil from 0% HOAc	Phosphorus mg/g of meats	mg/g of oil
Room[c]	0	192 ± 0.5	100.0	169 ± 1.3	100.0	0.01	0.05
Room[c]	5	213 ± 2.3	110.9	185 ± 7.4	109.5	0.32	1.52
60 C	0	195 ± 2.6	100.0	184 ± 0.8	100.0	0.02	0.10
60 C	5	215 ± 3.8	110.3	195 ± 4.3	106.0	0.35	1.59

[a]Values represent means ± standard deviations from the means. No standard deviations are given where deviations were so small as to be essentially 0.
[b]Total lipid is all materials in miscella after evaporation of solvent (crude oil).
[c]Ca. 25 C.

TABLE II

Filtration rate[a]

Percent of HOAc in hexane	Void time of funnel[b]	Time 1st pass	2nd pass	3rd pass	Mean
0	6.3 ± 1.5	15.3 ± 5.5	20.7 ± 9.6	20.0 ± 7.0	18.7 ± 7.0
5	7.0 ± 1.0	10.0 ± 0.0	10.0 ± 0.0	10.3 ± 0.6	10.1 ± 0.3

[a]6 g of comminuted soy meats extracted with 18 mL of solvent for 2 min and filtered through a 30 mL Buchner funnel of medium porosity with reduced pressure.
[b]Values in sec represent means ± standard deviation.

thorough extractions with hexane/acetone/water, chloroform/methanol/water and hexane/acetic acid were accompanied by disintegration of intracellular membrane structure (9,10).

The advantage of producing 6% more neutral oil (Table I) when acidic hexane is used at normal oil mill extraction temperatures (60 C) is obvious. A 2000 ton/day plant could divert ca. 20 ton/day of neutral oil from the meal fraction to the oil fraction with acidic hexane. Also, it is obvious that there is a potential for energy savings, by producing as much oil with hexane/acetic acid extraction at room temperature as could be produced by hexane extraction at 60 C. And, by doubling the filtration rate of miscella from marc (Table II), it might be possible significantly to increase plant capacity and/or reduce energy consumption.

The possibility also exists that a more desirable meal would be produced with acidic hexane. Lipoidal material, especially phospholipids and that material more soluble in polar than in nonpolar organic solvents, contribute to off-flavors (e.g., bitter, grassy, beany) of soybean and other legumes (11-14). Oxidized phosphatidylcholines, specifically, cause some of the bitter taste (13). Hexane/acetic acid extracts ca. 15- to 30-fold more lipoidal phosphorus than does hexane alone (Table I). This represents ca. 46% of the phospholipids of soybean (13). Some of these lipids probably contribute to soybean off-flavors and their removal would produce a more desirable food product.

Problems that may be anticipated in the use of hexane/acetic acid for oil extraction are: the corrosive nature of the acid, recovery of the solvent, refining of the oil, residual acetic acid odor of the meal, and nutritional properties of the meal. However, acetic acid in hexane is less corrosive than may be expected. At room temperature, it did not affect stainless steel, Teflon, or polyethylene after 28 days contact, except for a slight swelling of the plastics (6). Also, hexane/acetic acid can be recovered as a low boiling azeotrope (15,16). And, observations of the refined soybean oil color (visual and photometric, A_{440}) showed hexane/acetic acid-extracted oil and hexane-extracted oil to be equally refineable. The effects of hexane/acetic acid on the nutritional properties and the deodorization of meal have been examined recently (17).

ACKNOWLEDGMENTS

The authors thank J.A. Bogatz for technical assistance and G.R. List for valuable discussions.

REFERENCES

1. Jacks, T.J., L.Y. Yatsu, and A.M. Altschul, Plant Physiol. 42:585(1967).
2. Yatsu, L.Y., T.J. Jacks, and T.P. Hensarling, Ibid. 48:675 (1971).
3. Yatsu, L.Y., T.J. Jacks, and T.P. Hensarling, JAOCS 51:169 (1974).
4. Yatsu, L.Y., and T.J. Jacks, JAOCS, 49:937(1972).
5. Hensarling, T.P., T.J. Jacks, and L.Y. Yatsu, JAOCS 51:166 (1974).
6. Jacks, T.J., L.Y. Yatsu, and T.P. Hensarling, JAOCS 47:222 (1970).
7. Singleton, W.S., M.L. Brown, and H.J. Zeringue, Jr., JAOCS 43:592 (1966).
8. Clark, J.M., Jr., Experimental Biochemistry, W.H. Freeman and Co., San Francisco, CA, 1964, p. 32.
9. Hensarling, T.P., L.Y. Yatsu, and T.J. Jacks, JAOCS 47:224 (1970).
10. Jacks, T.J., L.Y. Yatsu, and T.P. Hensarling, JAOCS 51:169 (1970).
11. Eldridge, A.C., in Soybeans: Chemistry and Technology, edited by A.K. Smith and S.J. Circle, AVI Publishing Co., Westport, CT, 1972, p. 150.
12. Smith, A.K., and S.J. Circle, Ibid., p. 339.
13. Smith, A.K., and S.J. Circle, Ibid., p. 79.
14. Sessa, D.J., and J.J. Rackis, JAOCS 54:468 (1977).
15. Horsley, L.H., in Advances in Chemistry Series, No. 6, Am. Chem. Soc., Washington, D.C., 1952.
16. Horsley, L.H., and W.S. Tamplin, Ibid., No. 35, 1962.
17. Hensarling, T.P., and T.J. Jacks, JAOCS 59:516 (1982).

[Received January 4, 1982]