A Rapid, Effective Method for the Reduction of Waxes in Sunflower Seed Oil

W.H. MORRISON, III, USDA, ARS, Richard B. Russell Agricultural Research Center, PO Box 5677, Athens, GA 30613

ABSTRACT

Four samples of sunflower seed were surface washed with boiling hexane to remove waxes and hydrocarbons. Measurement of waxes in oil extracted from washed and unwashed seed showed greater than 92% removal of waxes. In addition, straight chain hydrocarbon content of the oil was substantially reduced. The composition of the material removed is shown.

Winterization of sunflower seed oil by conventional methods has presented problems due to the fine crystals produced and their tendency to become coated with mucilagenous materials which retard filtration (1). As an alternative, manufacturers have combined winterization with degumming (2) or cold alkali refining (3).

Reports have been received that oil from hybrid seed has a higher wax content than oil from open pollinated varieties. Studies have demonstrated that waxes are located principally, if not solely, in the hull, and that there is an inverse correlation between hull content of the seed and wax content of the oil and hull (4). The kernels in hybrid seed appear to be more tightly bound to the hull by fibrous material than in open pollinated varieties (4). The combination of these two factors could account for the higher wax content found in oil from hybrid seed. While complete hull removal would appear to be an alternative, this fibrous material is needed to maintain good extraction characteristics of the meal.

This report describes a rapid, effective method which would be incorporated into any prepress solvent extraction operation for the removal of over 90% of the waxes in sunflower oil.

MATERIALS AND METHODS

The four samples of high oil hybrid sunflower seed used in this study were: (a) Master Farmer 700 from Millhaven Co., Sylvania, GA, 1979 crop; (b) mixed hybrids from Arthur Farms, Arthur, ND, 1979 crop; (c) mixed hybrids from Interstate Seed Co., Fargo, ND, 1981 crop; and (d) mixed hybrids from the University of Florida, Gainesville, FL, 1978 crop. All samples were stored at -20 C when received and 0 C after cleaning until used.

Wax removal was accomplished by placing 100 g of seed in a Buchner funnel containing no filter paper. A 300-mL portion of boiling hexane was poured as rapidly as possible into the funnel, being careful not to cause an overflow and still covering seed. Under these conditions, the seed were in contact with the hot hexane for four seconds. Preliminary experiments showed that when 100 g samples of seed were washed with 100-, 200- and 300-mL portions of boiling hexane, the amount of material removed with 200 and 300 mL were equal. In addition, when 100 g of seed were placed in a beaker of 300-mL boiling hexane for 1.5 min, no more material was removed than when the seed was washed with 300 mL of boiling hexane. The oil content

TABLE I

Wax Content of Sunflower Seed Oil Before and After Hexane Washing

Sample	Wax content (ppm) Unwashed seed	Wax content (ppm) Hexane-washed seed	% Wax removed	Cold test (days)
1	885	68	92.4	2
2	1444	68	95.3	2
3	778	46	94.3	3
4	893	72	92.1	2

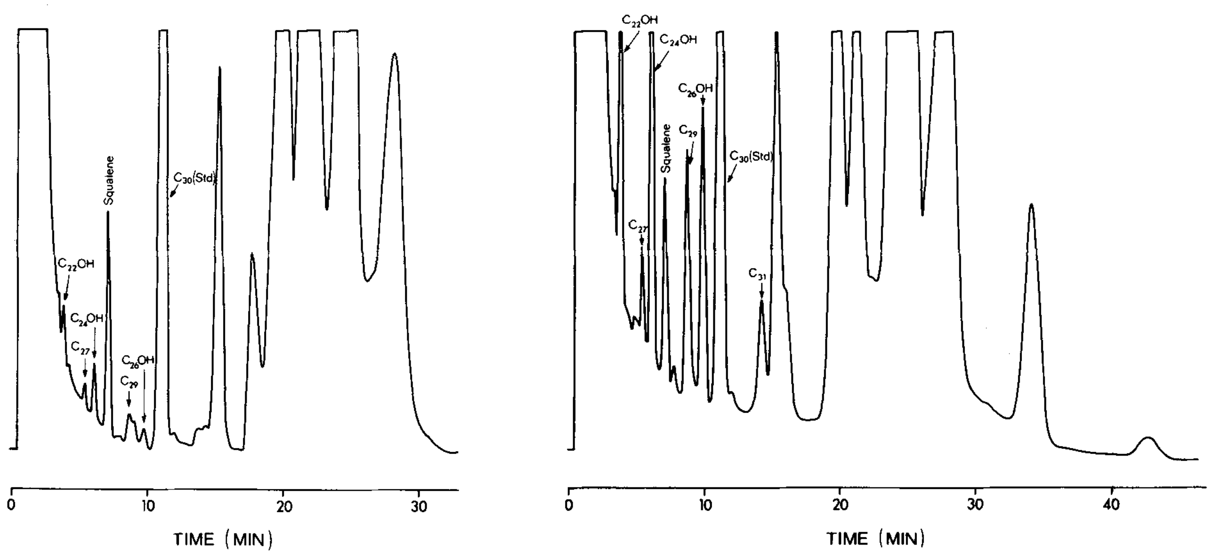

FIG. 1. Gas liquid chromatographic trace of hydrocarbons, sterols, and BSA derivatives of waxy alcohols in oil from (a) hexane washed sunflower seed, and (b) unwashed seed.

TABLE II

Composition of Waxy Residue Removed from Sunflower Seed with Hexane Washing

	Alcohols (%)					Hydrocarbons (%)			% Wax	% Hydrocarbons
Sample	C-20	C-22	C-24	C-26	C-28	C-27	C-29	C-31	in residue	in residue
1	12.3	30.3	28.7	17.8	11.0	8.5	50.1	41.8	69.7	30.3
2	22.4	38.6	21.3	11.1	6.5	12.2	50.3	36.8	72.7	28.3
3	17.8	36.9	24.2	13.6	7.5	11.2	51.7	37.0	87.0	13.0
4	16.0	35.3	24.4	13.6	10.6	12.5	50.4	37.0	79.0	21.0

of the sample before and after this treatment was identical, suggesting that no oil is extracted even under this extended contact with solvent. The filtrate was filtered through Whatman 41 filter paper while hot to remove suspended material and then transferred to a tared flask. After evaporation of solvent, the flask was reweighed to obtain the weight of the material removed. The surface washed seed were placed in a shallow pan to evaporate traces of solvent. The washed seed were ground in a Krups 75 mill and extracted in a soxhlet apparatus for 6 hr with hexane. After evaporation of solvent, a portion of the oil was evaluated by gas liquid chromatography for wax (5). In this procedure, the amount of C-22, -24, and -26 alcohols (chromatographed as the silylethers) is calculated using triacontane as an internal standard. The percentage of wax can then be determined based on the concentration of these alcohols. A second sample to be evaluated for a cold test was heated to 130 C, filtered, and sealed in clear glass bottles (5). The samples were refrigerated at 0 C and examined daily for clarity. At the first sign of loss of clarity, the day was recorded. Each hybrid was analyzed in triplicate.

RESULTS AND DISCUSSION

The effect of hexane washing on the wax content of the oil extracted from the seed is shown in Table I. The initial was content of the oil from unwashed seed ranged from 770 to 1444 ppm wax. Simply washing the seed with hot hexane reduced these values to between 46 and 72 ppm, resulting in over 90% removal of waxes. These results clearly demonstrate that waxes associated with sunflower seed oil are found almost entirely on the surface of the hull.

The cold test results shown in Table I show that three of the samples remained clear for two days and one for three. The loss of clarity was apparent only as a slight loss of brilliance. Only after several days could a thin layer of white precipitate be seen in the bottom of the containers with oil from hexane-washed samples.

Typical gas liquid chromatographic traces of waxy alcohols, hydrocarbons, and sterols in the oil from washed and unwashed seed are shown in Figures 1a and b. The major peak remaining in that portion of the chromatogram where waxy alcohols and hydrocarbons elute is squalene (5), which is apparently not found on the surface of the hull. As can be seen, hexane washing provides effective removal of hydrocarbons as well as waxes.

The waxy material removed from the seed by the hexane is a mixture of 70-87% waxes and 30-13% straight chain hydrocarbons. The composition of the waxy alcohols and hydrocarbons is shown in Table II. Hydrocarbon composition is fairly uniform. Alcohol composition also appears fairly uniform except for sample 2 where C-20 alcohol is slightly higher than C-24.

Preliminary studies indicate that a solvent to seed ratio (w/w) of between 1 and 2 to 1 was sufficient to remove optimal quantities of wax. It appears that this approach, used in combination with dehulling and conventional winterization procedures, would be an efficient approach for removing waxes from sunflower oil.

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