SOLVENT EXTRACTION OF CORN OIL FROM DISTILLERS GRAINS

By C. S. BORUFF and DWIGHT MILLER

Technical Director and Research Chemical Engineer
HIRAM WALKER AND SONS, INC.
Peoria, Ill.

Abstract

A process has been developed for extracting the corn oil from distillers' grains rather than the incoming corn. Yields by various methods of recovery are considered. Solvent extraction by the use of low boiling petroleum naphthas was adopted. Pilot unit extraction and refining was followed by oil acceptance by refiners. Feeding tests showed the extracted grains to be superior to the original distillers' grains.

The 100-ton extraction plant expects to be in full operation the early part of 1938.

At present, corn oil is produced chiefly as a by-product in corn products plants and in a very few distilleries, by expelling or hydraulic pressing the oil from the corn germ which has been separated from the whole grain by either the wet or dry milling process. Recently, at least one plant has equipped itself for solvent extraction of the corn germ. It is the opinion of the Hiram Walker Research Department that the corn oil should be left in the grain that is to be fermented and subsequently distilled to whiskies. Therefore, instead of considering the wet or dry degermination of the 15,000 bushels of corn used daily, consideration was given to the matter of recovering the corn oil following fermentation and distillation. To this end, experiments were made on methods of recovering the oil from the dried distillers' grains recovered after distillation, and which in the Hiram Walker plant represents 100 per cent of the residual grain material left after fermentation and distillation inasmuch as screens and centrifuges are used to recover the insolubles, and evaporators to recover all the solubles in the still discharge. By solvent extraction of the dried distillers' grains, it is possible to recover practically all of the corn oil present in the original grain, and it is necessary to work with only 35 to 40 per cent of the total weight of grain brought into the plant inasmuch as 60 to 65 per cent has been removed during fermentation. These dried grains consist chiefly of fat 6-8 per cent, protein 30 per cent, ash 3-4 per cent, moisture 10 per cent with the remainder being fiber and nitrogen free extract. They will weigh approximately thirty pounds per cubic feet.

In Table I are summarized representative corn oil yield data obtained by expeller and solvent extraction methods. As noted in this table, when the original whole corn contains from 3 to 4 per cent oil, through solvent extraction methods one may recover an average of 1.7 pounds of corn oil per bushel of grind as compared with .5 to .75 pounds by expelling oil from the germ recovered by the dry milling process and 1.25 pounds as recovered from the germ by the wet degermination process. Solvent extraction leaves less than 1.0 per cent oil in the residual grain, as compared to 5.0 to 6.0 per cent when the oil is removed from the germ by expellers.

TABLE I.

YIELD OF CORN OIL BY VARIOUS PRODUCTION PROCESSES*

Method of Recovery	Pounds of Oil per Bushel of Whole Corn
Dry degermination process...	0.5 to 0.75**
Wet degermination process...	1.25**
Solvent extraction of residual grain	1.5 to 1.9

*Whole grain containing 3 to 4% oil.
**Vegetable fats and oils — Jamieson, 1932, p. 151.

The solvents investigated included numerous low boiling petroleum naphthas and various chlorinated hydrocarbons. Due to the superior quality of crude oil obtained through the use of certain low boiling fractions of petroleum naphthas, the freedom from dangers to the personnel due to toxic solvent vapor poisoning,* and the comparative low price and unlimited resources, these solvents were adopted as preferable over the chlorinated hydrocarbons, in spite of the lower explosive hazards noted for the latter. Practically all large continuous extraction plants in operation at the present time use a petroleum naphtha solvent.

*Volatile Solvents, J. Am. Medical Assoc., Vol. 109, No. 10, Sept., 1937, p. 762.

Numerous methods of extraction and refining of the crude oil were investigated in the laboratory but discussion of these results will not be considered in this paper because the commercial scale investigation is more significant and gave results predicted by laboratory tests. One point of significance, however, bears consideration. Using the Official Methods of Oil Refining as published by the American Oil Chemists' Society laboratories we noted refining losses on this crude corn oil, possessing a free fatty acids content of 8.0 per cent, to range from 30 to 50 per cent, depending upon the quantity of alkali used, rate of stirring, temperature, time of settlement, etc. When this same corn oil was refined in the plant using Sharples equipment, the total refining and handling loss was noted to be only 18 per cent and this will probably be further lowered when larger than experimental quantities are treated. This indicates that there should be a new standard method of assay for such oils high in free fatty acids which have been obtained by solvent extraction of oil bearing materials.

After extensive laboratory tests indicated the soundness of proceeding with this investigation, pilot unit studies were completed, and then arrangements made for extraction of sixty tons of distillers' grains in a commercial size solvent extraction plant. After removal of phosphatides by centrifuging, the crude oil was collected for refining.

The extracted feed was also recovered and returned to Peoria for examination and testing purposes. In order to determine the feed value, as well as to check the possibility of any toxicological effect of the extracted grains on animals, Poll Angus calves of an original weight of 358 to 388 pounds were fed by the laboratory staff. The feed was composed of 23 per cent distillers' grains, 26 per cent ground corn, and 51 per cent alfalfa hay. In the one case, the distillers' grains was the unextracted feed containing 8 per cent fat, while in the second case, the distillers' feed contained only 0.5 per cent fat as determined by Soxhlet extraction using petroleum ether as the solvent. The feed containing the unextracted distillers' grains gave a gain in weight of 2.05 pounds per day while the feed containing the extracted distillers' grains gave a gain of 2.33 pounds per day. The calves were fed at the rate of 10.1 pounds of total feed per day. It required 4.9 pounds of feed per pound of gain when feeding the unextracted feed but only 4.3 pounds of feed per pound of gain when feeding the extracted grains. The analysis of the extracted and unextracted distillers' grains is given in Table II. The removal of oil increases the percentage of protein in the distillers' feed with a corresponding increase in feeding value and tendency to give solid flesh.

These extracted grains show no off taste or odors after standing for over eight months, but rancid odors and off tastes develop in the unextracted feeds within a few weeks, especially if the feed is ground.

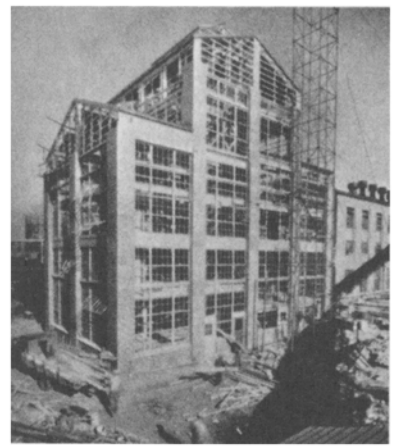
An appreciable quantity of the extracted feed was shipped to regular distillers' feed customers and was found satisfactory.

Part of the crude corn oil recovered during the plant scale run was sent to a commercial vegetable oil refiner who refined the oil, using their standard corn oil refining process, consisting of the Sharples alkali refining system and a final deodorization under vacuum with superheated steam. The oil was bleached slightly after alkali refining to remove some of the color before deodorization. Very little bleaching was done, and it may be possible to omit this procedure completely when refining in tank car lots.

Analyses of the crude and completely refined and recovered deodorized oil are given in Table III. It is noted that this oil is very dark and possesses a free fatty acid content of 8 per cent. The petroleum naphtha soluble color is due to certain processes the mash and grain have been subjected to in the distillery and feed recovery departments. It is removed readily by alkali treatment. The oil with its free fatty acid content of 8 per cent gives a 30 to 50 per cent refining loss when standard laboratory refining methods are used, but as stated before in this paper, a 5,000 pound batch of this oil gave a total refining, bleaching, deodorizing, and handling loss of only 18 per cent. Some of the refined oil was used in the manufacture of mayonnaise and other food products and some of it was sold in bulk to consumers. All reported satisfactory results and acceptance. The completely refined oil has been found by the Walker laboratories, as well as the laboratories of prospective customers, to be equivalent in stability, odor, taste, and chemical analysis to the better class of refined corn oils now on the market.

On the basis of the above, Hiram Walker and Sons, Inc., of Peoria, Illinois, is now building a solvent extraction plant to handle 100 tons per day of distillers' feed. Approximately, 14,000 pounds of crude oil will be recovered per day from this plant. This crude oil will be centrifuged to remove phosphatides, but will not be refined by Hiram Walker, but rather sold to vegetable oil refiners.

The engineering and construction plans for the extraction plant have been handled by the Engineering Department of Hiram Walker and Sons. All milling, extraction, drying, distilling, and solvent recovery apparatus is being furnished by Allis-Chalmers. The Bonotto extraction column is being used. The extraction plant should be in operation the early part of 1938.



Solvent Extraction Plant, Hiram Walker and Sons, Peoria, Ill.

TABLE II.
ANALYSIS OF EXTRACTED AND UNEXTRACTED DISTILLERS' GRAINS USED IN FEEDING TEST

	Moisture %	Protein %	Fat %*	Ash %
Extracted grains	5.9	32.5	0.5	3.5
Unextracted grains	5.9	30.7	8.0	3.2

*Petroleum Ether Solubles.

TABLE III.
ANALYSIS OF EXPELLED CORN OIL AS COMPARED WITH WALKER'S CRUDE AND COMPLETELY REFINED CORN OIL

	Expelled Corn Oil		Walker's Extracted Corn Oil	
	Crude[1]	Refined[2]	Crude	Refined
Specific Gravity	.921 to .927	.921 to .927	.92	.924
Refractive Index (15° C.)	1.477	1.475 to 1.477	...	1.476
Saponification number	187 to 193	187 to 193	186	191
Free Fatty Acids, %	1.3 to 2.0	.020	8.0	0.030
Color	13Y-.1R[4]	35Y-3.5R[3]	21Y-12R[4]	35Y-3.7R[3]
Odor	Poor	Good	Poor	Good
Taste	Poor	Good	Poor	Good
Kreis test	.....	Negative	.....	Negative

[1]Vegetable Fats and Oils—Jamieson, 1932, p. 154.
[2]Food Analysis—Woodman, 3d Edition, 1931, p. 184.
[3]Standard Cell—5¼" oil depth.
[4]Laboratory standard—1 part Oil, 5 parts clear hexane—¼" cell (Lovibond).