Effect of Moisture on Grinding of Tung Kernels and Solvent Extraction of Meal*

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IT IS a matter of common knowledge in the tung oil industry that the amount of oil which can be expressed from tung meal varies with the water content of the meal. The complete extraction of oil from tung kernels is known to be difficult. Even for laboratory analysis it is necessary to re-grind and re-extract meal after the preliminary grinding and extraction. In industrial processing it is obviously desirable to grind kernels adequately before extraction, so that practically all of the oil can be extracted in one operation without any additional grinding or handling. In the introduction of a solvent process for the extraction of tung oil it is of interest to determine the effect of moisture on grinding meal in preparation for such a process and to determine the effect of moisture in the meal on extraction of the oil.

In order to study these effects samples covering a wide range of moisture content were examined by the regular analytical technique (1) employed at the U. S. Tung Oil Laboratories. It was appreciated at the outset that with this technique the methods of extraction in industrial processing (2) would not be duplicated, and the comminution of tung kernels and consequently extraction efficiency, as measured by the proportion of oil extracted without re-grinding and re-extraction, would be affected appreciably by moisture content. While the method of grinding used in this procedure is not the same as that which may be used in industrial practice, it was considered that data of interest would be obtained and would provide a basis for predicting the effects of moisture in an industrial solvent process.

In determining quantitatively the amount of tung oil in meal samples, 5 grams of tung kernels ground in a No. 1 Russwin food grinder with a 16-tooth cutter were rolled in filter paper and extracted (1) in a Butt tube over a 150-ml. boiling flask for four hours. The sample was then removed and ground vigorously in a mortar with one gram of analytical-grade sea sand, and extracted for two more hours in a second 150-ml. flask. Concentration of the contents of both flasks was performed by distillation of the bulk of the solvent at ordinary pressure, followed by heating at 106°C. in a vacuum oven at 28½ inches of mercury. The sum of the weights of oil in the two flasks was taken as the total amount of oil extracted, and the percentage of the total amount of oil represented by the oil in the first flask was considered to be the efficiency of the extraction under the existing conditions of moisture and grinding. Moisture content was ascertained by determining the loss of weight after heating a 5 gram sample at 106°C. for 2½ hours in a vacuum oven at 28½ inches of mercury.

TWO solvents, as indicated in Table 5, were selected for use in these extractions. Skellysolve B is essentially a normal hexane fraction, boiling at 63° to 70° C., and Skellysolve F is essentially a pentanes-hexanes fraction, boiling at 35° to 60° C. Both of these solvents can be evaporated from tung oil with ordinary laboratory equipment with a minimum effect on oil quality (3). Generally, higher extraction efficiencies were obtained with the higher-boiling Skellysolve B than with Skellysolve F. This may be attributed to the removal of a substantial part of the moisture in the meal sample during the early stages of the refluxing in the extraction process by the solvent of higher boiling range. Since industrial extraction processes, generally, do not employ refluxing of solvent during extraction, this effect would be of value industrially only to the extent that moisture might be vaporized through heating the solvent to aid extraction.

TABLE 1
Comparison of Oil Extractions of Undried and Vacuum Dried Meal Samples. (Extractions With Skellysolve F)

Sample No.	Undried Meal			Meal Dried in Vacuum Oven for 2½ Hrs. at 106°C. and 28½" Hg.		
	Oil Extracted at End of		Efficiency (Proportion oil extr'd by initial extr'n)	Oil Extracted at End of		Efficiency (Proportion oil extr'd by initial extr'n)
	Initial Extr'n	Final Extr'n		Initial Extr'n	Final Extr'n	
	Pct.	*Pct.*	*Pct.*	*Pct.*	*Pct.*	*Pct.*
1	9.9	34.2	29.0	17.4	33.6	51.6
2	13.9	41.1	33.8	25.7	39.9	64.5
3	20.8	47.0	44.3	32.0	47.2	67.8
4	27.1	47.5	57.0	32.2	47.3	68.1
5	21.8	46.8	46.5	34.7	47.2	73.6
6	21.3	47.3	45.1	32.7	47.7	68.5
7	24.0	48.9	49.0	35.2	47.9	73.5
8	28.6	48.1	59.4	35.3	47.2	74.9
9	29.5	48.8	60.4	34.6	48.0	72.1

In order to facilitate preliminary examination of samples of a wide range of moisture content, quantities of tung meal were adjusted to various moisture levels as a supplement to naturally existing moisture levels, found in available tung fruits. Subsequently, examinations were made on kernels from the new 1943 crop. In analyses of material of different age and treatment, it soon became apparent that all data could not be compared on the same basis.

In order to study the effect of drying meals in these preliminary examinations duplicate samples were weighed. One of the duplicate samples was extracted without change in moisture content, and the other was extracted after drying in a vacuum oven for 2½ hours at 106°C. under reduced pressure of 28½ inches of mercury. (See Table 1.) Moisture contents of meal samples are not shown in Table 1, since this table is intended to indicate only the increase in extraction efficiency that may be obtained in the case of a given sample, when it is vacuum dried. Extraction efficiencies were appreciably greater on the dried meal than on the undried meal, indicating that the presence of moisture in the meal will sensibly affect the solvent extraction of the oil to the extent

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that only a portion of the oil freed by grinding will be obtained.

At the outset of this study only kernels of low moisture content were available, therefore, a quantity of kernels from stored tung fruits of the 1942 crop was ground and portions of the meal were allowed to stand over water in a tightly closed can for various periods of time to produce material of increased moisture content. The results of extracting four portions of such meal in comparison with the meal in its original moisture condition will be found in Table 2. It

TABLE 2

Extraction With Skellysolve F at Moisture Levels Produced by Allowing Meal Samples to Stand Over Water in Closed Can

Moisture in Meal	Oil Extracted at End of		Efficiency (Proportion oil extracted by initial extraction)	Time Required for Moisture-Conditioning Sample
	Initial Extraction	Final Extraction		
Pct.	*Pct.*	*Pct.*	*Pct.*	*Hrs.*
4.0	51.2	55.2	92.7	(Used in original condition)
12.3	45.6	50.8	89.8	12
12.8	46.0	51.2	90.0	44
14.6	47.0	50.2	93.6	68
16.8	41.7	47.0	88.8	72

was noted that the ground material had a rather low moisture content, and that the proportion of oil removed by the initial extraction, that is, the extraction efficiency, was relatively high compared with extraction efficiencies obtained on other meals in this study which came from the 1943 crop of tung fruit. It is reasonable to assume that this difference was due to the age of the material. It was also noted in this case that moisture added artificially had relatively little effect on the extraction efficiency; rather the state of comminution, which was effected at the 4.0 percent moisture content before moisture conditioning, seemed to be the predominant factor.

Another lot of kernels of fairly high moisture content was ground and allowed to dry after being spread in a thin layer on a laboratory table. Samples were withdrawn at various periods and analyzed as indicated in Table 3. It was noted here also that the extraction efficiencies did not differ one from the other to any very great extent for the decreasing moisture contents down to 3.8%, and it was considered that the grind accomplished on the material before drying had the greater effect on the oil extraction.

TABLE 3

Extraction With Skellysolve F at Moisture Levels Produced by Allowing Meal to Dry in Air

Moisture in Meal	Oil Extracted at End of		Efficiency (Proportion oil extracted by initial extraction)	Time Allowed for Drying
	Initial Extraction	Final Extraction		
Pct.	*Pct.*	*Pct.*	*Pct.*	*Hrs.*
25.4	28.3	48.5	58.3	(Used in original condition)
19.1	29.9	54.4	55.0	24
3.8	36.6	63.6	57.5	48

Other moisture levels were obtained (See Table 4) by placing samples of ground kernels in wide-mouth bottles, connected in series in such a way that air drawn through them would contact substantially all of the material. Air drawn through the samples was dried by first passing through calcium chloride and then through concentrated sulfuric acid.

TABLE 4

Extraction With Skellysolve F at Moisture Levels Produced by Passing Dried Air Through Meal Samples

Moisture in Meal	Oil Extracted at End of		Efficiency (Proportion oil extracted by initial extraction)
	Initial Extraction	Final Extraction	
Pct.	*Pct.*	*Pct.*	*Pct.*
34.3	17.1	43.0	39.7
32.8	19.0	45.1	42.2
31.5	18.6	43.1	43.1
31.1*	20.0*	46.7*	42.9*
28.0**	20.9**	46.7**	44.7**
27.5*	21.1*	47.1*	44.8*
27.2*	20.8*	47.1*	44.2*
26.5*	22.7*	47.9*	47.3*

*Average of 2 values.
**Average of 3 values.

IN AN examination of extraction data, however, it was noted that proportions of oil obtained in the initial extraction period with samples having an adjusted or conditioned moisture level were not in agreement with those obtained with samples of approximately the same moisture content, which were analyzed on an "as received" basis without any adjustment of moisture content in the laboratory. The conditioning of samples of tung kernels, frequently ranging from 60 to 70 percent oil content, presents a difficult problem after grinding because of the extrusion of appreciable quantities of oil on the meal surface, which undoubtedly inhibits moisture absorption and causes most of the moisture to be taken up at the surface. Accordingly, conditioned samples of tung meal should not be expected to compare closely in moisture distribution with unconditioned samples, even though over-all moisture contents of the samples are the same. It is possible also that part of the differences may have been due to slight oxidation and polymerization of surface oil during the moisture conditioning of samples. These differences present an interesting problem for further consideration.

Since in actual commercial practice tung fruits, and consequently, tung kernels, are handled on an "as received" basis (with fruits having been subjected to varied moisture conditions while on the ground and in open-side storage bins), it was felt that examination of such material over a considerable period of the storage season and from a number of widely separated orchards in the Louisiana-Mississippi area would yield information having a direct bearing on commercial operations. Data (see Table 5) obtained on such material with no laboratory moisture adjustment indicated that extraction efficiencies increased with decreasing moisture content until a limiting moisture level was reached, after which extraction efficiencies decreased with decreasing moisture content. It seems probable that this trend was due in large part to the state of comminution produced at the various moisture levels, and that the state of comminution obtainable was dependent to a considerable extent upon the moisture content. At high moisture contents a relatively poor grind was obtained with increasingly better grinds being obtained as the moisture content decreased. However, at approximately 6 to 9 percent moisture content peak extraction efficiencies were reached, and thereafter extraction efficiencies generally decreased with decreasing moisture content. It would seem, so far as the technique employed is concerned, that moisture serves as a grinding aid to a certain extent, and that

TABLE 5

Extraction at Moisture Levels in Meals "as Received"

Moisture in Meal	Skellysolve F: Oil Extracted at End of: Initial Extr'n	Skellysolve F: Oil Extracted at End of: Final Extr'n	Skellysolve F: Efficiency (Proportion oil extr'd by initial extr'n)	Skellysolve B: Oil Extracted at End of: Initial Extr'n	Skellysolve B: Oil Extracted at End of: Final Extr'n	Skellysolve B: Efficiency (Proportion oil extr'd by initial extr'n)
Pct.	*Pct.*	*Pct.*	*Pct.*	*Pct.*	*Pct.*	*Pct.*
47.8	8.8	27.1	32.6	9.4	26.8	34.9
47.2	9.8	32.6	30.2	11.3	30.5	37.2
46.6	9.1	29.8	30.4	10.7	29.7	35.9
37.7	11.7	35.0	33.5	14.0	35.6	39.2
33.5	13.7	39.3	34.9	19.3	40.3	47.9
33.1	12.3	35.2	35.0	15.6	35.5	44.0
27.8	27.0	47.5	57.0	......	......	......
24.6	28.3	48.5	58.3	......	......	......
23.0	26.5	48.6	54.5	36.4	49.7	73.4
19.0	30.3	51.2	59.1	39.7	51.6	76.9
17.6	31.5	56.0	56.3	41.1	56.7	72.6
16.4	29.4	49.5	59.5	36.0	50.2	71.7
15.8	36.8	54.7	67.2	41.5	53.0	78.3
14.9	35.5	52.9	67.0	43.8	53.1	82.5
14.0	34.8	59.4	58.6	41.0	59.4	68.9
13.3	39.2	55.2	71.0	46.6	56.4	82.5
12.4	43.6	55.9	78.0	49.2	56.8	86.6
12.2	40.3	61.7	65.3	48.2	61.7	78.2
9.9	43.3	62.0	69.9	50.7	61.0	83.1
9.1	47.1	62.4	75.4	50.2	62.3	80.6
8.7	45.3	63.1	71.9	55.1	62.8	87.8
7.9	47.3	62.2	76.0	56.3	63.3	89.0
7.1	54.1	65.1	83.1	58.9	64.7	91.0
6.7	50.5	59.5	85.0	54.5	59.9	91.1
6.6	51.4	65.2	78.9	58.8	64.6	91.0
5.6	51.2	64.5	79.3	57.2	64.1	89.3
5.5	51.9	63.3	82 1	56.3	63.4	88.8
5.4	50.6	64.4	78.7	58.8	65.1	90.3
5.4	53.5	65.0	82.3	60.6	65.1	93.2
5.2	53.4	65.7	81.2	57.0	65.5	87.0
4.9	52.0	67.6	77.0	55.2	67.6	81.7
4.0	50.4	65.8	76.6	54.6	65.6	83.3
4.0	49.6	66.5	74.6	52 9	66.9	79.0
3.3	51.9	67.1	77.4	......	......	......
3.3	49.9	68.0	73.3	......	......	......

there is an optimum moisture content for grinding kernels most effectively.

To obtain further data on this subject, portions of tung kernels containing 5.0 percent moisture were adjusted to various moisture levels, up to 14.6 percent, by addition of the requisite amount of water to each portion, placing it in an air-tight container and shaking occasionally until the water was absorbed and evenly distributed. One set of these samples was ground and analyzed in the usual manner, using the Russwin food grinder, while the other set of samples was ground in a Raymond attrition pulverizer. The resulting meals were extracted, reground and re-extracted, in the usual manner. The results of the extractions are shown in Table 6. It is interesting to note that with both grinding procedures the highest extraction efficiency was obtained when the moisture content of the unground kernels was in the neighborhood of 7 percent.

TABLE 6

Extraction With Skellysolve F at Moisture Levels Produced by Adding Water to Kernels Before Grinding

A. Using Russwin Grinder *

Moisture in Kernels	Oil Extracted at End of: Initial Extraction	Oil Extracted at End of: Final Extraction	Efficiency (Proportion oil extracted by initial extraction)
Pct.	*Pct.*	*Pct.*	*Pct.*
5.0	49.6	63.9	77.5
6.6	55.4	63.9	86.7
8.0	54.3	63.4	85.7
9.5	52.3	62.1	84.2
11.5	49.4	61.1	80.8
13.3	44.4	59.1	75.2
14.6	42.6	58.3	73.1

B. Using Raymond Pulverizer *

Moisture in: Kernels	Moisture in: Meal	Oil Extracted at End of: Initial Extraction	Oil Extracted at End of: Final Extraction	Efficiency (Proportion oil extracted by initial extraction)
Pct.	*Pct.*	*Pct.*		*Pct.*
5.0	4.5	62.8	63.2	99.2
6.6	5.2	64.8	65.3	99.2
8.0	6.6	63.5	64.3	98.9
9.5	7.5	63.0	63.8	98.9
11.5	9.1	61.8	62.7	98.6
13.3	10.7	60.1	61.1	98.4
14.6	12.1	59.1	60.3	98.0

* Identification of the equipment used by giving the name of manufacturer should not be construed as an endorsement of such equipment by the U. S. Department of Agriculture.

Summary and Conclusions

DATA are presented to show the effects of moisture on grinding of tung kernels and on solvent extraction of oil from the resulting meal. They indicate that:

(1) The efficiency of oil extraction can be materially increased by vacuum drying ground kernels before extraction.

(2) The state of comminution obtainable on grinding tung kernels is dependent upon moisture content; above 9 percent moisture content the efficiency of grinding, and consequently efficiency of extraction, decreases progressively with increase of moisture content; the most efficient grinding is obtained with material of moisture content in the range of 6 to 9 percent; and, in general, progressively poorer grinding is obtained on tung kernels with moisture contents ranging downward from 6 percent.

LITERATURE CITED

(1) McKinney, R. S., and Freeman, A. F., Oil & Soap *16*, 151-152 (1939).

(2) McKinney, R. S., Rose, W. G., and Kennedy, A. B., Ind. Eng. Chem. *36*, 138-144 (1944).

(3) Freeman, A. F., Pack, F. C., and McKinney, R. S., Ind. Eng. Chem. *35*, 1156-1159 (1943).

Smalley Foundation Report 1943-1944

WE are presenting herewith the 26th report of the Smalley Foundation Committee of the American Oil Chemists' Society. During these past 26 years considerable progress has been made in the accuracy of the determination of oil and nitrogen on cottonseed meal. The results obtained in practically all determinations were slightly lower than last year. It must be understood, in gauging the accuracy of the results, a difference of two points in either direction from the average is permitted without a deduction from the grade. We might add that the results obtained are so nearly perfect that a few hundredths of a per cent higher or lower than on any previous year means very little as far as accuracy is concerned.

During the year one of the collaborators called our attention to the fact that he obtained differences if he made corrections for the blank obtained on the paper and solvent used for extraction. He suggested that we poll the collaborators to find out how many of