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The relation of moisture content to the maximum extraction of oil from soybean flakes.

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The Relation of Moisture Content to The Maximum Extraction of Oil From Soybean Flakes

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INTRODUCTION

Scientific research, to speak generally, may have two motives. In one, the motive is the desire to extend the boundaries of knowledge; the other may have a special purpose of solving problems related to industry. However, the latter may reveal new knowledge while solving a problem in the factory. Industrial research is concerned not only in discovery of truth, but also the production of something useful; and which in addition, yields profit. The intention of technical research is to create new industries or to develop old industries into higher or more productive forms.

This paper is not the result of an attempt to reach deep into theoretical chemistry but simply the report on the study of an industrial problem. With the introduction of solvent extraction to meet the greater demand for soybean oil, many problems arise as to the conditions for maximum oil yield. This problem is an attempt to determine what moisture in soybean flakes will yield the maximum oil when extracted by hexane and petroleum ether. These solvents were used because hexane is commonly used in commercial extraction and petroleum ether is employed in the control laboratories.

This problem becomes increasingly important at this particular period due to a shortage of vegetable oils and fats. Their relation to the food and explosive industry make it imperative that every effort be made to extract as much available oil as possible. No important literature was found on the subject. In truth, all literature found that considered the moisture as a factor was a report of experiments performed on ground soybeans. A. A. Horvath, (3) who has spent many years in the soybean industry in Asia and the United States, is of the opinion that soybeans with 12 per cent moisture will give the maximum yield of oil. Soybean oil processors have held the same opinion. The object of this research is to determine if a lower moisture content of soybean flakes will yield more oil by solvent extraction.

HISTORICAL

The production of vegetable oils and fats occupy a place among the world's greatest industries, since their final products are consumed by man wherever he exists. A perfect history of the development of the industries may be obtained by a study of the various process methods in present use throughout the world. In countries where little or no industrial progress has been made, primitive methods of obtaining oil still survive. The most primitive of methods (2) is exposure to heat from the sun as in the case of the cocoanut. Not many years ago the Japanese allowed fish to rot in heaps until the oil exuded. One of the earlier methods of extraction (4) was to crush the seeds and boil the meal with water, the oil rising to the top and being skimmed off from time to time. In later methods the seeds were crushed and pounded in mortars. The earliest form of oil press was known as the wedge press. The seeds were placed in bags of horse hair, which were placed in tanks or boxes and pressure applied by driving a wedge between two bags. This method of pressing oil from soybeans is at present performed in native Chinese mills. These were superseded by screw presses, similar to a wine press, and later by hydraulic presses of various types. The chief disadvantage of the older methods was the high residual oil content of the cake. The expeller method was developed to meet the need of a continuous operation. This method, similar in principle to that of a meat grinder, when once started, requires much less labor and attention.

In recent years a greater demand for oil has necessitated the removal of a higher percentage of the total oil since the cake from pressure methods contains from 5 to 10 per cent oil. To meet this need, solvent extraction has been developed. The extraction of oil by solvents is of a comparatively recent origin since it dates from 1843 (1) when the large scale manufacture of carbon bisulfide made solvent operation possible. The present day extension of the solvent process is due entirely to the great improvement in methods of preparing and refining solvents. Formerly applied to low grade and waste materials only, the process is now used in the extraction of oils of the highest quality.

MATERIALS AND METHODS

All experiments were performed in the laboratory of a soybean oil separation plant. The proximity of the place of experiments to the plant afforded an easy access to all materials needed.

A. Materials

(1) Soybean flakes of .005 inch in thickness.

(2) Extraction apparatus consisting of Butt extraction tubes, reflux condensers and soxhlet flasks.

(3) Copper extraction tubes.

(4) Analytical balance.

(5) Solvents of petroleum ether and hexane.

(6) Hot water bath.

B. Methods

It is the opinion of the writer that the methods were entirely new, inasmuch as no literature was found on the extraction of oil from flakes. Nor were any methods found that poured solvent over the flakes as in the extractor column of an extraction plant.

The methods may be divided into four groups.

(1) The soxhlet extraction method:
Five grams of flakes at different moisture contents were extracted for three hours with petroleum ether.

(2) The soxhlet extraction method:
Five gram samples of flakes at different moisture contents were extracted for three hours with hexane.

(3) Copper tube extraction method:

Ten gram samples of flakes at different moisture contents were extracted with 100 ml. of petroleum ether in 20 ml. portions.

(4) Copper tube extraction method:

Ten gram samples of flakes at different moisture contents were extracted with 100 ml. of hexane in 20 ml. portions.

EXPERIMENTAL

Approximately thirty sets of experiments including one hundred fifty samples were extracted by the four methods previously mentioned over a period of four months. Flakes of known moisture content were split into several portions. The flakes were .005 inch in thickness and were collected over a twenty-four hour period. The desired moisture was obtained by adding water or drying slowly in an air oven.

A - In the Soxhlet extraction method, employing petroleum ether as a solvent, 5 gram samples of flakes were placed in filter thimbles and inserted in Butt extraction tubes. They were refluxed for three hours on a hot water bath. The flasks were heated to a constant weight after evaporation of the solvent and weighed to determine the per cent oil.

B - Duplicate samples were treated as above with the exception that hexane was used in place of petroleum ether. The results are shown in Tables I and II and in the accompanying graphs. The per cent oil extracted is shown at the various moisture content of the flakes and also shown converted to a dry basis. It was necessary to convert to a moisture free basis for an accurate comparison of the dry material used in each sample. There was little difference in the amount of oil extracted by the two solvents, the hexane yield being slightly higher, but oil yields with both solvents increased as the moisture was decreased.

TABLE I

Soxhlet Extraction Method

Three Hours With Petroleum Ether

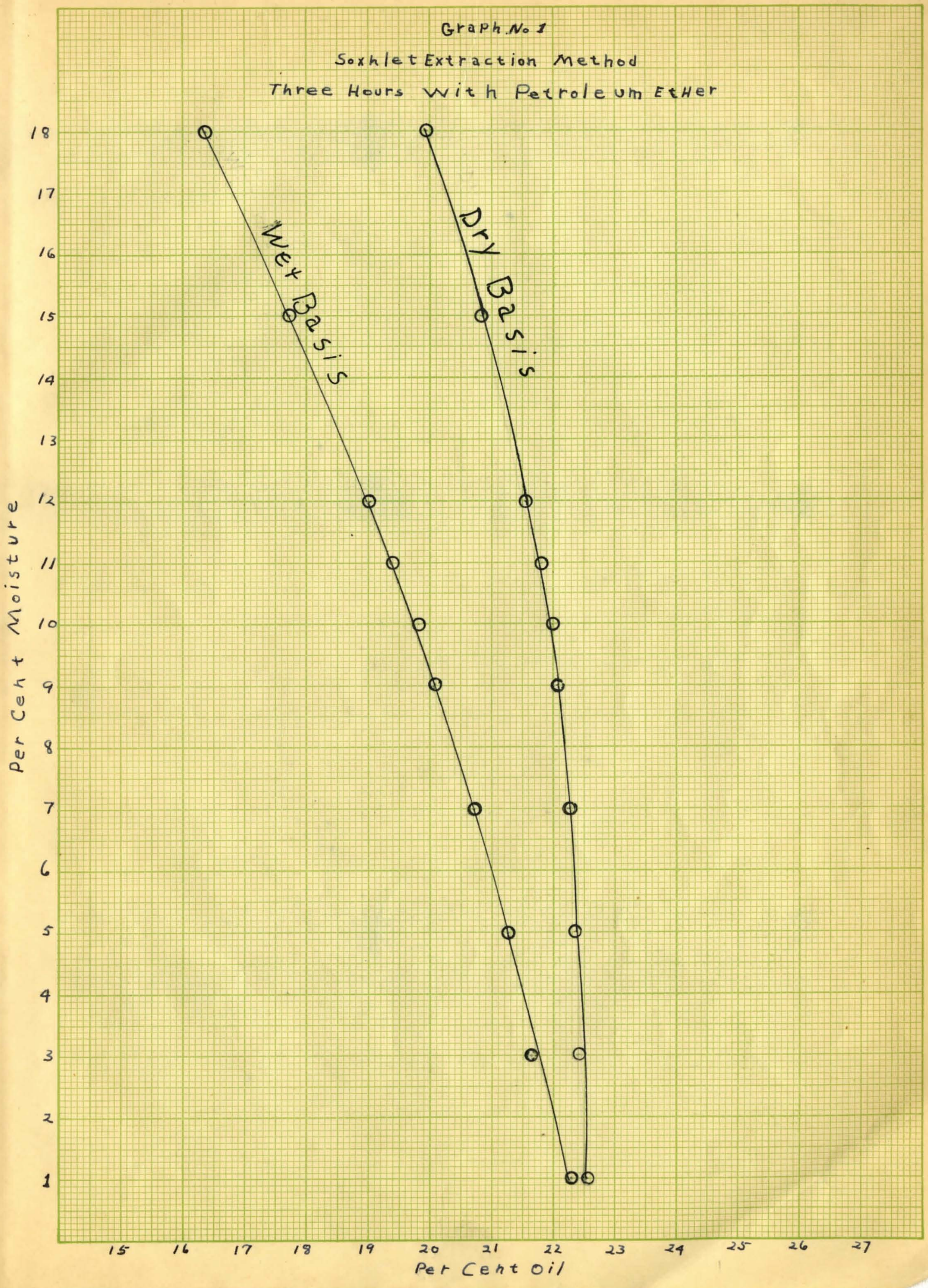
Sample No.	% Moisture	% Oil (Wet Basis)	% Oil (Dry Basis)
1	1.0	22.30	22.53
2	3.5	21.62	22.40
3	5.0	21.23	22.35
4	7.0	20.70	22.26
5	9.0	20.08	22.04
6	10.0	19.80	22.00
7	11.0	19.40	21.80
8	12.0	19.00	21.59
9	15.0	17.70	20.82
10	18.0	16.38	19.97

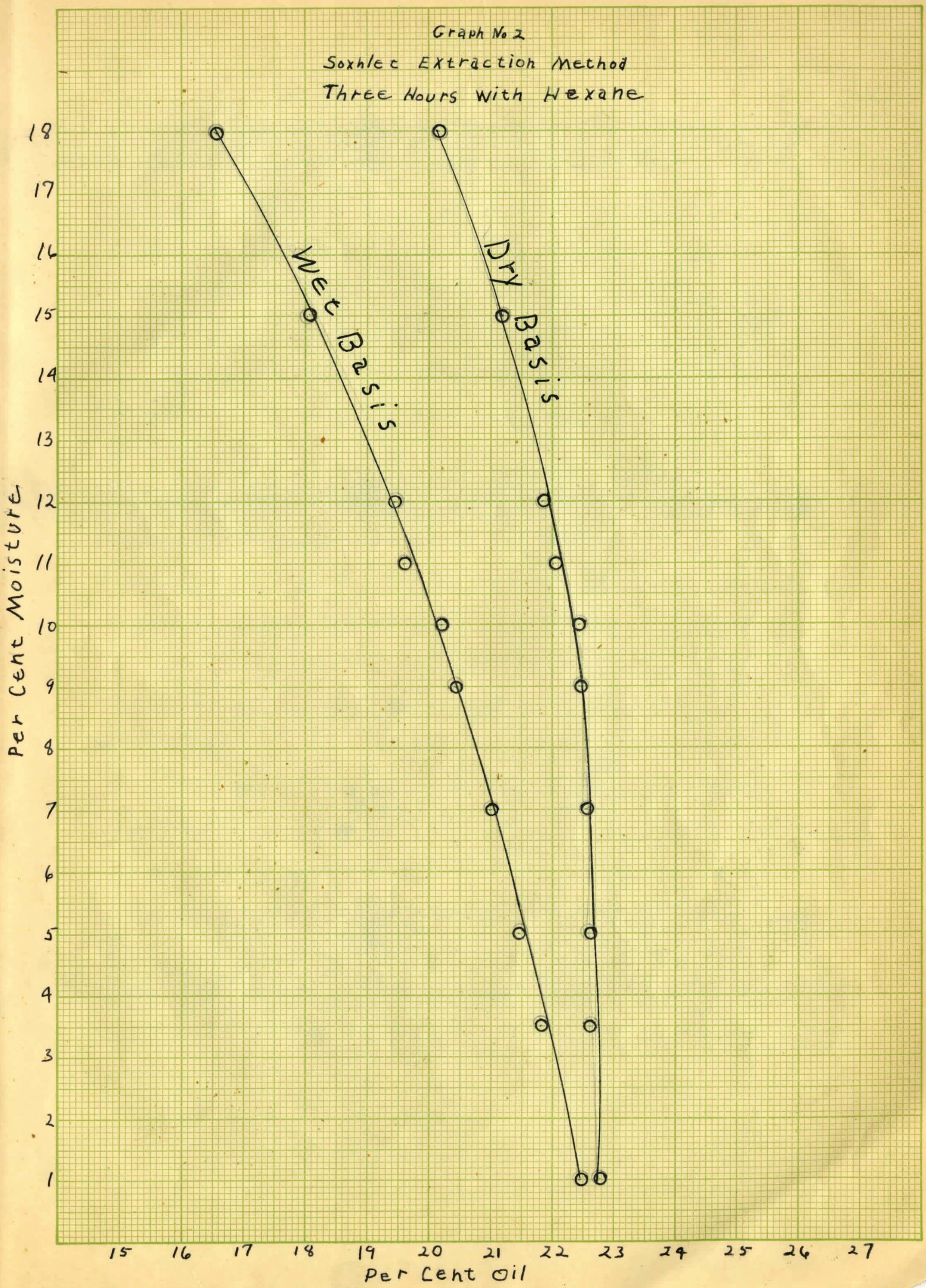
TABLE II

Soxhlet Extraction Method

Three Hours With Hexane

Sample No.	% Moisture	% Oil (Wet Basis)	% Oil (Dry Basis)
1	1.0	22.48	22.71
2	3.5	21.81	22.60
3	5.0	21.47	22.60
4	7.0	21.00	22.58
5	9.0	20.42	22.45
6	10.0	20.20	22.44
7	11.0	19.60	22.02
8	12.0	19.45	21.85
9	15.0	18.02	21.20
10	18.0	16.52	20.15


Graph No 1
Soxhlet Extraction Method
Three Hours with Petroleum Ether
Wet Basis
Dry Basis
Per Cent Moisture
1
2
3
4
5
6
7
8
9
10
11
12
13
14
15
16
17
18
15
16
17
18
19
20
21
22
23
24
25
26
27
Per Cent Oil
AMERICAN PAD & PAPER CO., HOLYOKE, MASS.


Graph No 2
Soxhlet Extraction Method
Three Hours with Hexane
Wet Basis
Dry Basis
Per Cent Moisture
18
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
15
16
17
18
19
20
21
22
23
24
25
26
27
Per Cent Oil

The solvent extraction plant is a continuous operation, therefore, soxhlet extraction is not commercially practical. In the extraction column, the solvent is poured over the flakes as they rotate in baskets. The copper tube extraction method was devised to simulate, as nearly as possible, the industrial method of extraction.

C - The copper extraction tube method is very simple in procedure. In this case, 10 gram samples of soybean flakes were placed in copper tubes with a filter in the bottom. The 100 ml. of petroleum ether were heated and poured over the flakes in 20 ml. portions and allowed to drain for 30 minutes into soxhlet flasks. The solvent was then evaporated on the water bath until the weight of the flasks were constant.

D - Duplicate samples were treated the same as in the previous method, with the exception that hexane was the solvent.

The results are shown in Tables III and IV and in accompanying graphs on both wet and dry basis. This "pour over" method, as it may be termed in later discussion, gives a slightly lower yield of oil than the reflux method, but there is a greater increase in per cent oil extracted with a decrease in moisture. This method yields less oil because the solvent is in contact with the flakes for a short time only.

TABLE III

Copper Tube Extraction Method

By Pouring 100 ml. of Petroleum Ether Over Flakes

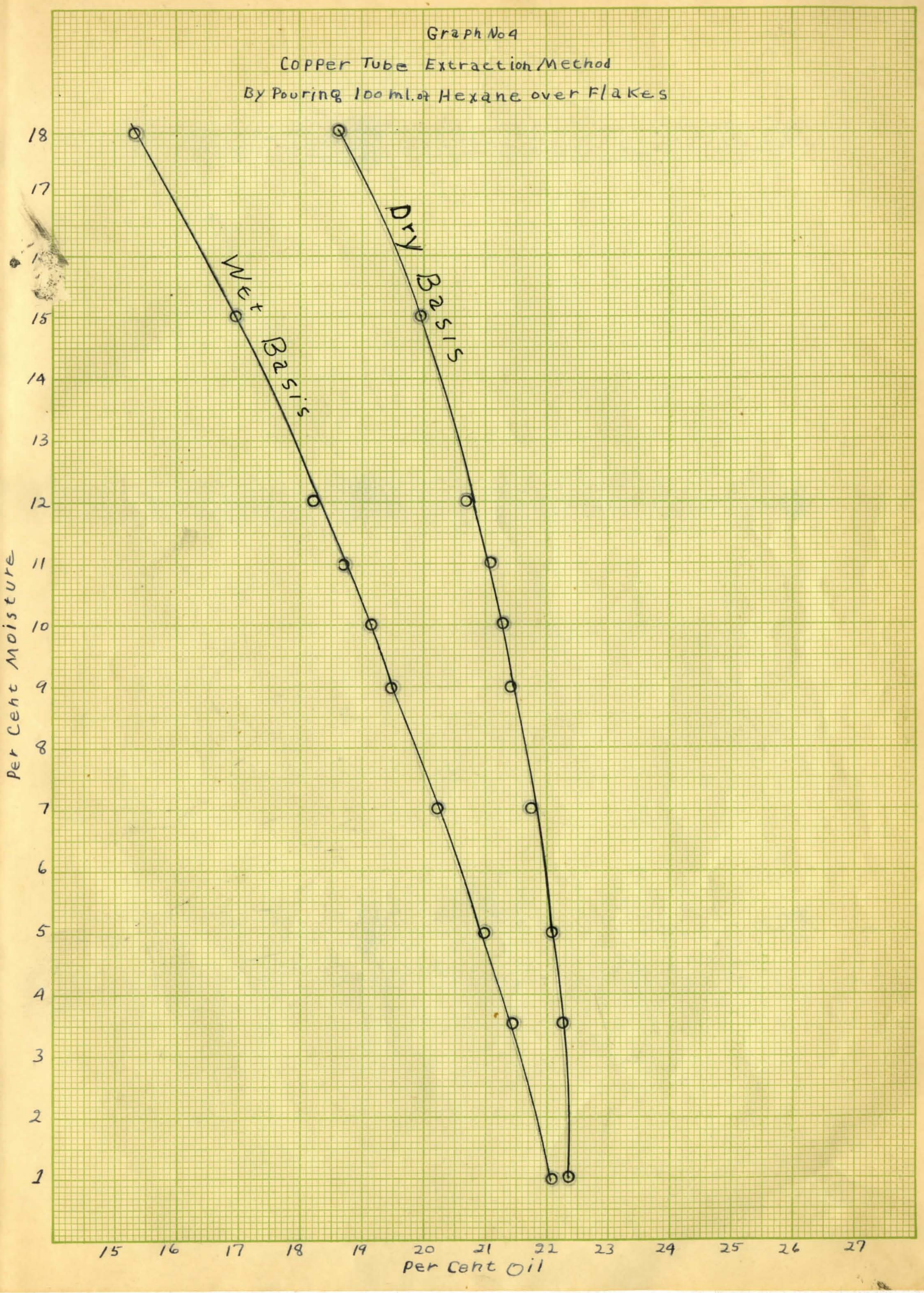
Sample No.	% Moisture	% Oil (Wet Basis)	% Oil (Dry Basis)
1	1.0	22.20	22.42
2	3.5	21.50	22.28
3	5.0	21.06	22.17
4	7.0	20.35	21.88
5	9.0	19.60	21.54
6	10.0	19.30	21.44
7	11.0	18.85	21.18
8	12.0	18.30	20.82
9	15.0	17.00	20.00
10	18.0	15.59	19.01

TABLE IV

Copper Tube Extraction Method

By Pouring 100 ml. of Hexane Over Flakes

Sample No.	% Moisture	% Oil (Wet Basis)	% Oil (Dry Basis)
1	1.0	22.07	22.29
2	3.5	21.37	22.14
3	5.0	20.90	22.00
4	7.0	20.14	21.66
5	9.0	19.45	21.37
6	10.0	19.10	21.22
7	11.0	18.70	21.01
8	12.0	18.20	20.68
9	15.0	16.95	19.94
10	18.0	15.32	18.68


Graph No 4
Copper Tube Extraction Method
By Pouring 100 ml. of Hexane over Flakes
Wet Basis
Dry Basis
Per Cent Moisture
Per Cent Oil
18
17
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
15
16
17
18
19
20
21
22
23
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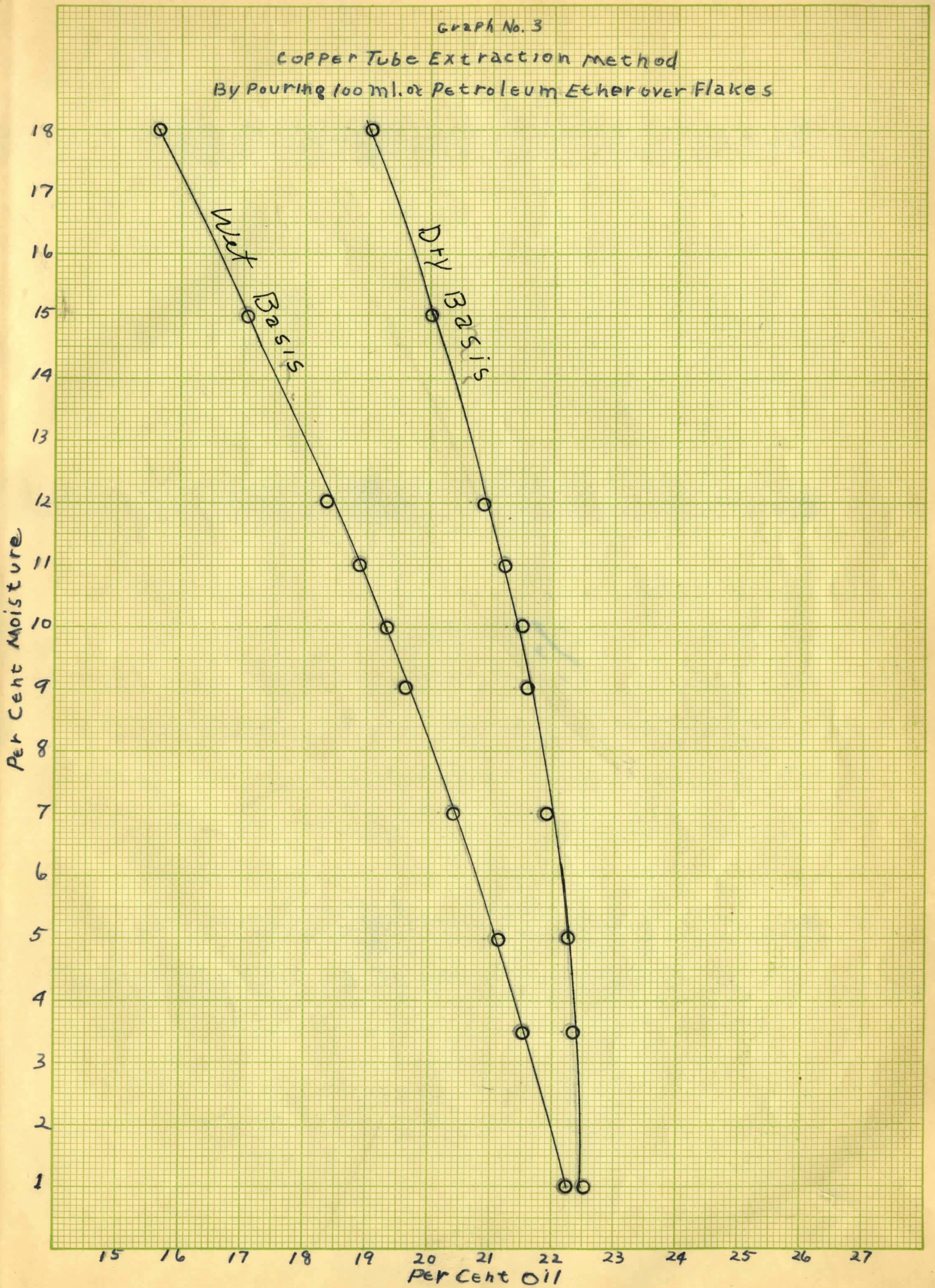
AMERICAN PAD & PAPER CO., HOLYOKE, MASS.



Graph No. 3
Copper Tube Extraction method
By Pouring 100 ml. of Petroleum Ether over Flakes
Wet Basis
Dry Basis
Per Cent Moisture
Per Cent Oil
18
17
16
15
14
13
12
11
10
9
8
7
6
5
4
3
2
1
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23
24
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27

DISCUSSION

It has been the general opinion in the soybean industry that soybeans should contain 12 per cent moisture for maximum oil extraction. All experiments in this paper showed that a 1 per cent moisture content gave the highest yield of oil when extracted by solvents of hexane and petroleum ether. These results prove that water is an inhibiting factor in the extraction of oil with hydrocarbons.

From the atomic linking structure of a compound, one may predict its solubility behavior in a qualitative way. Hexane is only slightly soluble in water (1:1000) (5) because of the dissimilarity in structure between hydrocarbons and water. From this fact, we can see how the presence of water would have an inhibiting effect on the extraction of oil with a hydrocarbon. In the extraction of oil from a substance that contains moisture, the solvent should have a higher boiling point than water. If this solvent were then heated and poured over the flakes, the water would be vaporized, allowing the solvent to penetrate the cells more readily. Hexane has a boiling point of 148-155° F. which is considerably lower than water. This point is proved to a certain extent in the two types of experiments. In the prolonged reflux method of extraction, there is less variation in the oil yield than in the "pour over" method. By refluxing, the flakes are held at a higher temperature for a longer period of time, which probably vaporizes much of the water to permit better extraction.

Soybeans contain the phosphatides, lecithin and cephalin and it is quite possible that their presence affect the oil yield. The phosphatides are highly hygroscopic and may shift into the aqueous phase in an excess amount of moisture. Since the quality of work done by the extractor column is based on three hours extraction of extracted flakes in the laboratory, this would give a higher residual oil in the flakes; because the phosphatides held by the water would be extracted over a three hour period of refluxing. With a lower moisture, the phosphatides remain in the oil. Their presence then lowers the surface tension of the oil and results in a wetting of the flake surface with oil. In some extraction methods, phosphatides are added to the solvent as a wetting agent to give added power of penetration.

Part of the moisture in soybeans may be in loose chemical combination, but it is probable that a large part of it is adsorbed by the hydrophilic groups of proteins and carbohydrates. When these groups adsorb moisture, they enclose oil droplets surrounded by water and prohibit prnetration by the solvent.

The solvent extraction process has the advantage of extracting more available oil than pressure methods. In order to hold this advantage, the solvent plant must be kept at top capacity to prevent loss in time, labor and solvent expense. It was found that beans containing 12 per cent moisture must be dried to permit better cracking and flaking. In this process, the flakes are fairly hot when they leave the flaking rolls, and in transit to the extraction building, they are cooled to a certain extent. Even after the hot air is drawn off, there is some condensation of moisture on the flake surface and in small air pockets. This "sweating" process greatly decreases the effectiveness of the solvent in extraction of oil.

In addition to an increased oil yield, it is probable that a lower moisture content in soybeans also increases the tonnage of beans extracted during a given unit of time. This is very important from the industrialist point of view. By lowering the moisture from 12 to 10 per cent, there is a marked increase in tonnage. It is not practical to crack and flake beans of a much lower moisture content than 10 per cent, but it is possible to dry the beans after flaking. As far as the writer knows, this has not been tried commercially.

The results of this research showed that flakes of 1.0 per cent moisture content gave the highest yield of oil. The moisture free flakes did not always give higher results than 1.0 per cent moisture flakes. This probably was due to oxidation of the oil caused by excessive heating. Polymerized compounds of high molecular weight exhibit decreased solubility in inert solvents. The process of oxidation or "drying", which is not fully understood, may be a process of polymerization. It is also quite possible that a molecule of water may enter the double bond forming hydroxy acids. In this case the presence of several hydroxyl groups along with the ester unions would decrease the solubility in hydrocarbons. Flakes may be dried to a 1.0 per cent moisture content by heating gently and thereby preventing oxidation. Further evidence of the effect of moisture may be noted in the color of the oil; the more highly colored oil being extracted at the lower moisture contents. Since one of the chief phenomena associated with the ethylenic linkage is the occurrence of color, it would seem that water has the greater influence on the unsaturated oils. That the color of many compounds is an outcome of structure, is shown by the fact that when the ethylenic bond is broken by the addition of hydrogen, the color disappears. If brought in contact with water and certain lipases, vegetable oils hydrolyse readily and split apart into glycerol and the fatty acids. Since the presence of fatty acids is undesirable, especially if the oil is to be used for food, it is better to extract oils under conditions least favorable for their formation and which will yield the most oil. With materials such as soybeans, which have a comparatively low oil content, profitable operation becomes difficult unless a high per cent of the oil can be removed.

SUMMARY

Two types of experiments were performed in the extraction of soybean oil from flakes. One method was extraction by refluxing for three hours and the other by pouring the solvents over the flakes. Two solvents, hexane and petroleum ether, were used in both methods. The following conclusions are based on the results:

1. The solvents used in both methods gave the maximum yield of oil when the soybean flakes contained 1.0 per cent moisture.
2. The reflux method gave a slightly higher yield of oil than the "pour over" method.
3. Hexane gave higher yields by refluxing and the petroleum ether yields were higher in the "pour over" method.
4. The yield of oil in each case decreased with increased moisture.

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