

LOW BOILING

HYDROCARBONS

AS OIL EXTRACTION MEDIA

By H. ROSENTHAL and H. P. TREVITHICK

The use of solvents for the extraction of oils from oleaginous materials has long been recognized by chemists and chemical engineers as having many advantages over the expression methods now used by the vegetable oil industry, particularly because the yield of oil in solvent practice closely approaches the theoretical. This increases the value of the products by the difference in value of the recovered oil as meal and as crude oil. Unit labor costs moreover are considerably lower in solvent extraction plants than in hydraulic procedure, while press cloth expense in the latter generally exceeds the cost of solvent losses in the former. Little headway has been made in the introduction of present solvent extraction processes in this country however, in spite of the known advantages of such processes as to potential yields of oil and adaptability to bulk production methods. This has been due to several defects in the present extraction processes, as now known and practiced which have contributed to limiting the commercial usage of these processes in this country.

The following are prominent among these defects:

1—The crude oil obtained in these processes was frequently of dark color containing much dissolved foreign matter from the meats of the oil bearing seeds, and was generally inferior in quality. This was particularly noticeable in extracted cottonseed oil which required more and stronger lye for refining than expressed oil, and which would not produce prime refined oils without excessive losses in refining. In addition solvent losses were often excessive because of incomplete removal of the solvent from the oil, or the temperatures used in removing the solvent were so high as to cause "setting" of the color in the oil.

2—The prices of solvents were relatively high, so that even moderate solvent losses resulted in high costs.

3—The residual meal produced often retained sufficient solvent or solvent odors to render them objectionable as cattle feeds.

Facilitates Extraction

All of these drawbacks to the success of solvent extraction processes may be obviated by the use as solvents of the low boiling hydrocarbons, particularly propane and butane, of which there exists a plethora in the United States. Let us consider in detail how the use of these solvents overcomes the various present objections to solvent extraction processes.

Since these solvents are gases at ordinary temperatures and pressures, they must be kept under increased pressure during the extraction operation in order to maintain them as liquids. This condition facilitates extraction at low temperatures, under which conditions, only the true fixed oils, free from all but the smallest amounts of gums, resins, coloring matters and other impurities, are removed from the extracted seed. As a result, in all the tests which have been made with these solvents, the crude oils—cottonseed, soyabean, peanut, etc.—have been very light in color and of good appearance, refining easily with low losses to very light colored refined oils. After complete removal of the solvent, the flavors were prime or better. Solvent removal is easily accomplished by reducing the pressure within the oil containing vessel to atmospheric levels or lower, with comparatively slight elevation of the temperature, with agitation, and with or without the use of open steam.

Similarly, because these solvents are gaseous at ordinary temperatures, the meal can be completely freed of all traces of the hydrocarbon by warming it in thin layers, as, for example, on a belt traveling in a current of warm air. The meal is then very light in color, of good texture and appearance, suitable not only for animal feeding but even for human consumption.

Very Limited

Both propane and butane are at present drugs on the market, considerable volumes of them being actually wasted daily. This situation is not a result of the depression, but of the requirements of the petroleum and natural gas industry. These two hydrocarbons can not be used in the natural gas for transmission because they are insufficiently volatile; conversely they can not be incorporated in the liquid petroleum products because their volatility is too high. The markets which the producers have been able to develop to date for these products being very limited in scope, the present price of either propane or butane is but little in excess of the actual production and refining costs. Thus, either material can be purchased for about 4c a gallon at the refineries or gasoline plants in Oklahoma and Texas, where are found a large developed supply and an enormous potential supply of both. The freight rates from these production points to the leading cottonseed crushing mills are low; in fact, in certain of the districts, the solvent supply will be adjacent to the crude mill, and in many others the solvent can be piped directly to the point of use.

No Important Change

There is no potential use in sight for either product which might be expected to make sufficient inroads into their supply to change these conditions markedly. If all the vegetable oil mills in the country were using these solvents, the amounts so used would not be noticeable in comparison with the quantities available. It is reasonable to expect that there will be no important change in the supply and price of these solvents. Egloff and Shaad have estimated, that in 1929, for instance, the availability of these two hydrocarbons as follows:

Propane—111,415,000,000 cu. ft. or about 3 billion gallons.

Butane — 64,980,000,000 cu. ft. or about 2 billion gallons.

Both of these products are gases at ordinary temperatures and pressures, but they are easily maintained in the liquid phase under moderately increased pressure. There is only one propane, but there are two butanes, normal and iso, usually found associated with each other. Some of the more important properties of these hydrocarbons are as follows:

	N-Butane	Iso-butane	Propane
Boiling Point °F.....	33	11	-43
Melting Point °F.....	-211	-229	-240
Spec. Grav. of vapor.....	2.007	2.007	1.523
Spec. Heat of vapor (Cp)....	.458		.475
Spec. Heat of liquid at BP...	.55	.52	.52
Heat Value (BTU/lb.).....	3274	3274	2519
Cu. Ft. of vapor per gallon..	32.5	32.5	38.2
Wt. per gallon of liquid—lbs.	4.8	4.8	4.25
Limits of inflammability as percentage by volume in air	1.9 to 8.5		2.4 to 9.5

Latent Heat BTU/lb. at atmos.	166	159	183	Free Fatty Acids	Loss	Color of Refined Oil	Caustic Used
Vapor pressure:				1.6.....	6.2	35 Yellow 4.1 Red	8.5% 12 Be
Lbs. per sq. in. ga. at 60° F.	11.6	24	92.4	2.5.....	6.7	7.0	10.3 12
80° F.	22.9	39.2	128.1	1.6.....	6.4	4.3	8.5 12
100° F.	37.5	59	172.0	1.6.....	6.4	4.3	8.5 12
120° F.	56.1	83.3	225.0	1.7.....	6.8	3.8	8.7 12
				1.6.....	6.8	4.1	8.5 12
				1.6.....	5.0	6.5	8.5 12
				1.1.....	3.6	8.8	7.5 12

Sources:

Oberfell and Alden—Natural Gasoline.
 Haslam and Russell—Fuels and Their Combustion.
 Dana Jenkins, Burdick and Timm—Ref. Eng. Vol. 12, No. 12.
 Pittsburgh Equitable Meter Co. Handbook.
 Philgas Co.

Most of the properties of these hydrocarbons have been thoroughly investigated and may be readily obtained from numerous sources so that no difficulty in designing equipment for their use is to be anticipated from the lack of knowledge of the properties of the solvents. Furthermore, the petroleum industry has had long experience in handling the huge volumes of these materials under conditions much more severe than any which would be encountered in vegetable oil extraction.

In making our experiments on these solvents, we have used a pressure extractor, or cylinder, with internal diameter of about 3 inches and 36 inches long, equipped with pressure gauges, thermometers, upper and lower feed lines and bypasses, and holding about 2,000 grams of meats. The solvent was stored in tanks under 20 to 200 lbs. pressure and fed directly to the charged extraction cylinder, through a measuring tank. Experiments were made with down flow and also up flow of solvent. Three washings were made on each charge, with a suitable quantity of solvent to fill the voids in the charge and to cover the meats, and the oil left in the meal was less than 1½%. The yield of oil was 97% or more of the theoretical possible. This oil, as mentioned before, is of light color, good appearance, and of a quality much different from that of usual extracted oils.

The following table shows the results of refining tests made on extracted cottonseed oils, refined according to the standard methods of the American Oil Chemists Society:

These losses are considered to be higher than would occur in practice, as the samples of oil still contained some meal and moisture and would be better clarified in plant practice.

By releasing the pressure, the solvent was changed to a gas again and thus removed from the extracted oil. However, in practice, agitation, and possibly the use of a little open steam would facilitate this. Under laboratory procedure, some of the oils retained traces of solvents, but others refined to a prime grade completely free from all traces of the hydrocarbon.

One test was made with the extractor charged with ground whole cottonseed. The resultant oil was also light colored as made, and refined to 35 Yellow 6.0 red with a loss of 4.2%.

One test was made by extracting cottonseed meats obtained from a crude oil mill, and comparing the extracted oil with the pressed oil made by the mill at the same time. The pressed oil gave a color of about 9.5 with 13.7 loss while the extracted oil refined to about 5.3 red with 11.7 loss.

In the laboratory the extracted meal was aired only, and in a short time was completely free of any taste or odor of solvent. The meal was light colored, soft, easily pulverized, and with the characteristic odor of cottonseed.

Other tests are being made on soybeans, peanuts, etc., as well as on packing plant materials.

It would seem that this process offers possibilities to the oil crushing industry which are very intriguing in these days of little or no profits, as it offers a method of increasing the oil yield at a low cost, with satisfactory results in the quality of both oil and meal. Any process which will increase the value of the products \$1.50 to \$2.00 a ton should prove very interesting.

THE CHICAGO CONVENTION

OCTOBER 11 AND 12

A meeting of the Chicago convention Committee of The American Oil Chemists' Society was called by Chairman Irwin at Swift & Co. on June 21st. It was definitely decided to hold this year's meeting in the Tropical Ball Room of the beautiful Medinah Club, 505 North Michigan Avenue, which is certainly one of the show places of Chicago. This Club was built by the Medinah Athletic Club absolutely regardless of expense. It is of Oriental architectural design and is furnished with Oriental splendor, tempered only to the point of assuring its guests of every modern luxury. The location is ideal, being just north of the loop, yet very convenient to the loop and the Fair, which takes it away from the crowded activities of ordinary hotel surroundings, and will give the entire convention a homelike and cozy atmosphere.

The Committee can assure those who stop at the Medinah Club of modern, clean and comfortable rooms, most of which give a marvelous view of Lake Michigan and of The Century of Progress, itself, and the guests are entitled to all of the privileges of the Medinah Club, which includes a very fine swimming pool, bowling alleys, and a gymnasium that is one of the finest in the country. In fact, no guest of the Medinah Club needs to go outside of the Club for every possible recreational facility.

At this committee meeting, which included Chairman Irwin, Dr. L. M. Tolman, A. E. King, A. W. Putland, A. A. Robinson and J. P. Harris, a very constructive program was discussed. Other members of the Committee are Dr. J. J.

Vollersten, M. L. Sheely, A. E. MacGee and Dr. R. C. Newton.

In this connection the Committee wishes to extend an invitation to every member of the Society to send in suggestions covering the program, and any one who wishes to volunteer to present a paper himself, or has a suggestion as to some good speaker for the program, will be more than welcome.

This fall meeting at the Medinah Club will make possible by far the best entertainment that the Society has ever enjoyed in Chicago, because the Club itself will provide every one with a very wide variety of entertainment features, and in addition to this there will be a special entertainment Thursday evening, which will consist of the Second Annual dinner of the Society at the Medinah Club, only this time it will be held in the beautiful Grill Room. This is said to be the most beautiful grill in Chicago. There will be music and dancing during dinner and after dinner, and for those who do not care to dance, there will be the second annual card party. The Fifth Annual Bowling Tournament of the Society will also be held on Thursday evening, under the direction of our able impresario, Al King, who promises both more prizes and a larger entry list even than last year. Alleys will be available to any of the members at any time they desire to use them.

It is also a pleasure to announce the Second Annual Golf Tournament at Chicago, and its great success can be forecast, because Dr. L. M. Tolman will act as chairman, and he is arranging this year to hold this tournament at one of

Chicago's sportiest courses, right in the heart of the city and only 20 minutes ride from the Medinah Club, which will undoubtedly make this the best golf tournament that the Society has ever held here. A fine lot of prizes will be offered and a fine time will be enjoyed by every one. The convention is held early enough this year to insure Chicago's very best weather, so come to the meeting prepared to participate in the Golf Tournament.

The exhibits will be installed in the meeting room, and very choice space will be available to those firms desiring to present their products favorably to the chemists and executives of the oil and soap industry. Diagrams of exhibition spaces will be sent out at an early date, and firms wishing space should send in reservations promptly to John P. Harris, treasurer of the Chicago Convention Committee, Room 1511, 205 W. Wacker Drive, Chicago.

Last but not least is the second edition of The Century of Progress, which is being held at Chicago again this year, and do not let anybody tell you that this is not a better World's Fair from every angle than last year's Fair.

It is predicted that this will be the best convention that has ever been held at Chicago, just as the last New Orleans meeting was the best convention ever held in New Orleans. Our arrangements at the Medinah Club will do away with such disagreeable features attached to holding the meeting at a hotel, as inability of the members to secure suitable hotel accommodations, etc., as was experienced last year. Certainly no member who can possibly attend this meeting should miss it. "Ask the man who attended last year," and then figure that it will be just twice as good this year, and you will get some idea of what is in store for you.