TABLE IV

Effect of Method of Removal of the Oil and Solubility of the Protein on the Apparent Viscosity of Cottonseed Meal Glue Mixes

Meal	Nitrogen, %	Lipids, %	Percent of total nitrogen soluble[a]	pH of glue mix	Viscosity poises[b]
					(1 hr.)
Hydraulic-pressed	6.6	5.6	20.2	12.9	322
Hydraulic-pressed	6.7	5.6	39.0	12.9	297
Screw-pressed	6.8	3.6	40.0	13.2	311
Screw-pressed	6.9	3.6	42.7	12.8	317
Screw-pressed	6.9	4.7	50.4	12.7	295
Hexane-extracted	9.0	1.5	80.0	12.4	320

[a] Percentage of total nitrogen soluble in a 0.5 M sodium chloride, 2.5 grams of meal suspended in 100 ml. of solvent for 3 hours at 25°C.
[b] Glue mix compositions: Cottonseed meal, 100 parts; sodium hydroxide, 4 parts; sodium silicate, 15 parts; carbon disulfide-carbon tetrachloride, 3 parts; calcium hydroxide, 15 parts; trichloroacetic acid, 1.25 parts; water, 335 parts. See Hogan and Arthur, J. Am. Oil Chem. Soc., *28*, 20-23 (1951).

addition of trichloroacetate ion to prevent gel formation and to improve their tackiness was previously reported (1, 2). The data presented in Table III show that the presence of trichloroacetate ion has a similar effect on the properties of cottonseed meal dispersions. The optimum amount of ion to add is about 1.3 grams of trichloroacetic acid per 100 grams of meal; when compared on a protein basis, this amount is about equal to the optimum quantity added to protein dispersions. It is seen that if an amount in excess of this quantity is added to the meal dispersion, a decrease in its viscosity is observed.

Effect of Method of Removal of the Oil and Solubility of the Protein Contained in the Meal. The apparent viscosities of glue mixes, prepared from cottonseed meals from which the oil has been removed by different processes, are compared in Table IV. An indication of the extent of denaturation of the protein contained in the meal, which takes place when the oil is extracted, is its change in nitrogen solubility. In these selected meals their nitrogen solubilities in 0.5 M sodium chloride solution varied from 20 to 80% of the total nitrogen. It is significant that there is no substantial difference in the viscosities of the glue mixes prepared as indicated. However, as noted in a previous publication, a decrease in the nitrogen solubility of the protein contained in the meal results in a decrease in the adhesive strength of the glue.

On comparing these data with those for isolated protein, it is observed that the viscosities of the protein dispersions are also affected by the method of the removal of the oil from the meats (3).

Summary

It has been shown that the apparent viscosities of cottonseed meal dispersions depend on the concentration of meal in the dispersion, the concentration of sodium hydroxide in the dispersion, and the addition of trichloroacetate ion and that the viscosities of the glue dispersions are independent of the nitrogen solubilities of the protein contained in the meal and, for the processes evaluated, independent of the method of removal of the oil to produce the meal.

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Better Oil From New or Old Deodorizers[1]

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THERE are at least seven factors influencing the product obtainable from a deodorizer. These are: condition of feed stock, condition of stripping steam, condition of equipment, materials of construction, method of heating, operating vacuum, and time of deodorization. Consideration must be given to all of these points in analyzing for imperfections in a deodorizing system. However it is not intended to discuss these factors in detail but to touch upon them lightly, with more emphasis upon several specific ideas which may prove helpful to a refinery operator or superintendent.

Condition of Feed Stock

To produce a high grade of deodorized product it is necessary properly to pre-condition the oil being fed to a deodorizing system. Refining and bleaching techniques are well known, and substantially complete elimination of soaps is a "must" for good deodorization. Most soaps are generally removed at the tail end of the refining operation, but subsequent bleaching is needed to insure a top quality final product. The bleaching operation does take out traces of soap which would otherwise be present after refining and which cannot be removed by deodorization.

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Condition of Stripping Steam

It is important that adequate stripping steam be used and that it have sufficient superheat to avoid cooling the oil being deodorized. Any marked reduction in oil temperature will reduce the effectiveness of deodorization by reducing the vapor pressures of the undesirables to be removed. In the author's opinion this does not mean that steam must enter the deodorizer at precisely the same temperature as the oil, or for that matter above it, since the amount of sensible heat required to raise the temperature of dry steam to the deodorization temperature is quite small. However it does mean that the steam must be completely dry, contain sufficient superheat to insure dryness throughout the piping system leading to the deodorizers, and enter the deodorizer at a temperature reasonably close to that of the oil batch. From a practical point of view, in a batch deodorizer with a well designed sparger, the blowing steam temperature may be 30°F. below the oil temperature without noticeable effect because the heat transfer from the oil to the steam is accomplished over a wide area. Due also to the immense steam volume it is impossible for a particular portion of the oil body to become chilled. On the other hand, the presence of moisture from wet steam requires a concentrated application of considerable heat through a limited heat transfer area.

A possible source of trouble insofar as oil taste is concerned lies in the impurities often present in industrial steam. Boiler compounds, if carried with the steam, can damage oil flavor to a marked degree. For this reason, any liquid entrainment in the steam lines leading to a deodorizer is a potential hazard to the maintenance of top oil quality. Air in blowing steam is another impurity damaging to oil flavor and stability. Some deodorizing systems are furnished with their own steam generators where plant steam conditions are bad.

Condition of Equipment

Under this heading a number of possibilities present themselves. A very important point in any deodorizing system is the need for absolute tightness of connections and fittings. A small air leak, even though the vacuum equipment adequately maintains a very low absolute pressure, may give an off-flavor to the oil. Such leaks usually carry more air than that present in stripping steam and consequently are more serious.

It is common practice among deodorizer operators periodically to check their systems for tightness when temporarily shut down. The test using ammonia vapor and a sulfur taper on a cold system is excellent since it shows the position of a leak. However it is quite feasible to make a careful check for tightness during operation under vacuum at full temperature by means of a new leak detector (1) now on the market. This has the advantage of also finding those leaks which open up as the deodorizing system is heated to operating temperature. It is interesting to note that the manufacturer of this detector has not yet seen fit to recommend its use in the manner which will be described, but by actual test the detector has been found useful for the work involved.

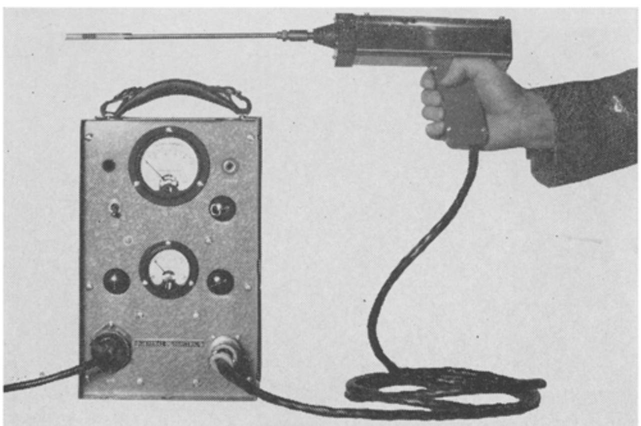

FIG. 1. G.-E. leak detector, "Type H." View showing detector unit (right), control unit (left), and leads.

Briefly, the leak detector, shown in Figure 1, consists of a milliammeter, a voltage regulator, a probe, and an element sensitive to halogen derivatives. Under ordinary circumstances, where vacuums are not involved, the portable probe with its small exhaust fan is placed near suspected points of leakage. Within the system there is maintained a moderate gas pressure with air and a halogen derivative. Usually the test gas contains Freon, which is readily available.

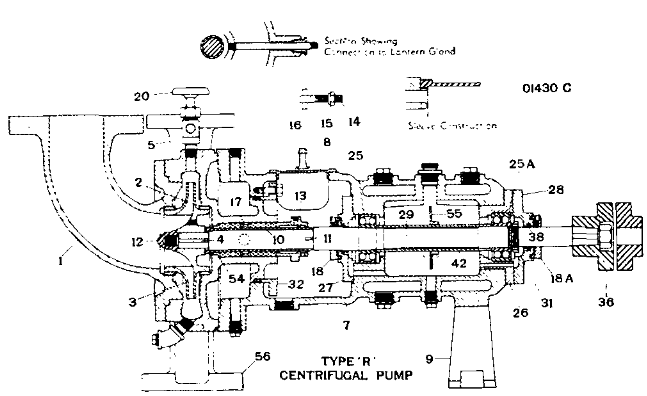

FIG. 2.

The probe picks up traces of Freon at a leaking joint, which in turn is blown over the sensitive element. In the presence of Freon vapors the element permits the passage of a relatively large amount of current which is registered on the milliammeter.

In a vacuum system the principle of leak determination is the same, except that test gas must be released outside of the equipment near the suspected leak and the probe placed in the vapor discharge of the vacuum system. It has been shown that this instrument can easily detect the presence of Freon in the discharge of a vacuum system even though diluted with steam from the vacuum air ejectors.

On account of its sensitivity care must be exercised in the use of this detector to insure true readings. For example, cigarette smoke near the probe quickly registers on the instrument, which reportedly has a sensitivity about one-tenth that of a mass spectrometer. Obviously its use would be nil in any plant where the atmosphere normally contains halogen derivatives, such as those used in some liquid heating systems.

Possibly due to accidental air leakage or mal-operation it is possible for a burned oil film to form inside a deodorizer. If this is not completely removed, the operator may expect degraded products as long as the film is present. In this connection considerable product improvement is likely if a methanol-caustic cleaning of the deodorizing system follows the usual caustic wash.

Air can sometimes enter the deodorizing system through stuffing boxes of valves and pumps. At critical points it is possible to install valves with extended stuffing boxes, which include lantern rings. The application of low pressure steam in these lantern rings eliminates the possibility of air leakage. Silicone lubricated valve stems are also employed.

In the case of the pumps the usual procedure calls for recirculating a stream of oil from the pump discharge to a lantern ring in the pump stuffing box. While this method is satisfactory in most cases, an alternate procedure has proven itself more reliable and convenient. This alternate method eliminates the need for a lantern ring and permits two or more additional rings of packing by sealing the stuffing box from the inside of the pump. A typical pump, and one which we have used with a great deal of success, is shown in Figure 2. It will be noted that this pump has a balanced impeller, by which it is meant that suction pressure exists on both sides of the impeller due to the presence of holes through the impeller. However, satisfactory pump operation does not require a balanced impeller and removal of these holes plus reasonable precautions taken by the pump manufacturer now makes it possible to apply a positive pressure from the pump discharge to the inner end of the stuffing box. This results in outward leakage of oil in place of inward leakage of air. With a well-aligned properly packed pump, oil drips from the pump may be limited to a few drops per minute.

Batch deodorizers are sometimes not as effective as they could be because of improper steam distribution in the base of the deodorizer. Consideration must be given to the amount of stripping steam involved as well as the available pressure. Uniform distribution of steam will assist deodorization.

Method of Heating

The easiest thing to say about vegetable and animal oils is that they are all different. Where you can effectively deodorize one oil at 425°F., you cannot get by with less than 450°F. with another oil. However when considering the possibility of burning an oil, one cannot take as a criterion the main oil body temperature. It is really the skin temperature of the oil in contact with the heating surface that is critical. Therefore it is very important that the type of heating and the heating medium be carefully considered in any deodorizing plant. Obviously the best heating medium is one which gives up its heat at a single minimum temperature and not over a temperature range. This will insure a minimum temperature on the surface of the heating element. Such satisfactory heating mediums include high pressure steam and Dowtherm, both of which give up latent heat and do not depend upon a temperature drop to provide sensible heat.

The importance of skin temperature rather than main oil body temperature was dramatized in one plant. It was found that the temperature range permissible for deodorization was extremely narrow, which made production of either under-deodorized or burnt oils a common occurrence. This condition was eliminated by increasing the oil velocity through a change to the oil heater flow pattern, thereby increasing the heat transfer rates. This, in effect, reduced the skin temperature of the oil, making it possible to produce a satisfactory product over a relatively wide range of main oil body temperatures rather than the narrow range previously necessary.

Materials of Construction

This subject is pretty well known in the industry. Ordinarily, correctly specified stainless steel and nickel are used successfully with little effect upon the oil. Carbon steel also is frequently a satisfactory material, but it must receive an oil film coating before satisfactory products are produced. For example, in a very old carbon steel continuous deodorizer it was found that product color improved through the first 24 hours of operation, a circumstance which proved the formation of the protective film.

Some batch deodorizing systems operating at temperatures between 400 and 500°F. make use of an external shell and tube heater in place of heating coils inside the deodorizer. Although carbon steel is used in the construction of heavy batch deodorizer coils, it should not be used for the tube bundle in a shell and tube heater during normal times. Carbon steel in this application will probably not last many months. Not only are the tubes relatively thin, but they must be subjected to a constant wiping action resulting from high oil velocity in the tubes. This high velocity is necessary to permit efficient heat transfer and low tube wall skin temperature.

Operating Vacuum

High vacuums are obviously beneficial to good deodorization by making it easier to strip out volatile impurities. In most parts of the country where summer water temperatures range from 80-85°F., three-stage vacuum equipment giving absolute pressures of 6-7 mm. Hg. are economically attainable. Where low water temperatures are available, the operator should look into the possibility of using better vacuums. For example, with 65°F. water, 3 mm. absolute pressure is quite feasible.

Time of Deodorization

The principal function of the deodorizer is stripping out of undesirables. In the batch process where time element may be varied, this time will depend principally on the ease with which impurities may be removed. In a plant handling any given oil there is considered to be a definite irreducible time cycle. There frequently is however a means by which the time cycle can be appreciably reduced. This is due to the fact that in most batch deodorizers the shell area above the liquid level is relatively cool, only being covered by layers of insulation. Therefore the condensation of distilled fatty acids and other undesirable volatile materials will take place on this cooled surface. By applying heat to the vessel area above the liquid level, it is possible to eliminate condensation, thereby eliminating the need for redistillation and thus decreasing the deodorizing time. A patent (2) has been issued to cover this operation, and it includes examples of a number of typical results to be expected by the application of "hot top" operation. As only one example of the use of deodorization with and without a "hot top," the following data are available as an illustration:

BATCH DEODORIZATION WITH AND WITHOUT "HOT TOP"

Top	Unheated	Hot
Blowing steam, lb. per hr.	64	64
Vacuum	10 mm.	10 mm.
Original F.F.A.	0.52%	0.50%
F.F.A. after 1 hr.	0.39%	0.35%
F.F.A. after 2 hrs.	0.36%	0.27%
F.F.A. after 3 hrs.	0.27%	0.21%
F.F.A. after 4 hrs.	0.22%	0.16%
F.F.A. after 5 hrs.	0.18%	0.11%

Of course, flavors are more difficult to evaluate than such positive factors as free fatty acid content. However flavor evaluation of many samples indicates that flavors and odors were removed during batch deodorization at about the same rate as free fatty acid removal. There are, of course, exceptions, but no accepted means of evaluating taste on a quantitative basis has been developed.

In conclusion, the generalization may be made that many deodorizing systems can be measurably improved if all factors influencing operation are carefully considered.

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