

TABLE No. I.

Crystallization of Saturated Glycerides from Acetone at 30°C.

Fat	I ₂ #	% Crystallization
Cottonseed Oil	0.0
Coconut Oil	0.0
Partially Hydrogenated Cottonseed Oil	66.7	0.0

TABLE No. II.

Solubility of Stearin in Acetone at 30° C.

Stearin	I ₂ #	Gms Added to 100 cc	Gms Soluble in 100 cc	% Solubility
Soya	2.19	1.5334 Gm	0.2310 Gm	15.05
Soya	20.94	1.6276	0.6822	42.00
Oleo	23.33	1.6257	1.1282	69.30

TABLE No. III.—Data for Fat Crystallized from Acetone at 30° C.

Fat Mixture PH C/S*	Stearin	Wgt. Stearin in 20 gms Fat	I ₂ #	% Solid Fat Recovered	Wgt. Solid Fat Recovered	I ₂ # Solid Fat Recovered	M.Pt. Glycerides	M.Pt. Fat Acids	Bomer Number**	Wiley M.Pt. Solid Fat Recovered
100.0%	0.0%	0.0000 gm	0.00	0.00%	0.0000 gm	0.0
99.0% Soya	1.0%	0.2056 gm	1.82%	0.3654 gm	11.04
97.0	3.0	0.6009	5.37	1.0754	9.80
95.0	5.0	1.0048	7.21	1.4435	7.59	67.0	63.7	73.6	66.2
92.0	8.0	1.6070	10.74	2.1492	6.03	68.0	64.3	75.4	66.9
0.0	100.0	2.19	68.9	67.8
99.0% Soya	1.0%	0.2047 gm	0.58%	0.1166 gm	11.60
97.0	3.0	0.6067	3.13	0.6264	10.38
95.0	5.0	1.0087	5.17	1.0352	9.98	66.0	62.4	73.2
92.0	8.0	1.6041	6.82	1.3643	9.00	66.9	63.0	74.7
0.0	100.0	20.94	64.5	62.9
99.0% Oleo	1.0%	0.2063 gm	0.01%	0.0034 gm
97.0	3.0	0.6291	0.16	0.0320	10.07
95.0	5.0	1.0102	0.38	0.0777	9.16
92.0	8.0	1.6119	2.26	0.4538	8.90	62.0	58.5	69.0
0.0	100.0	23.33	56.0	55.9

*Partially Hydrogenated Cottonseed Oil, I₂# 66.7.

**Bomer No. = M.Pt. Glycerides + 2 (M.Pt. Glycerides - M.Pt. Fat Acids)

Continuous Deodorization—Two Years Later

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1. INTRODUCTION

Two years ago we had the pleasure of presenting to the American Oil Chemists' Society a description of the process of continuous deodorization of edible oils, which was developed over a period of several years, and was represented at that time (in 1938) by the successful operation of one 5000 lb. per hour capacity unit and one 1200 lb. per hour unit shown in Figure No. 1.

Developments since that time have confirmed the economies and practicability of the continuous deodorization process, and the very marked saving in steam, fuel and condensing water requirements reflected in lower operating cost as compared to the usual batch method of handling.

2. OPERATION CYCLE OF CONTINUOUS DEODORIZATION VERSUS BATCH DEODORIZATION

Primarily, the process of deodorization consists of distilling out of the oil various volatile constituents which are undesirable, including residual free-fatty acids.

The release of volatile matter from the non-volatile glycerides is accomplished in continuous deodorization, just as in batch deodorization, by the combined application of heat and vacuum together with the introduction of injection steam, which, as in other distilling operations, reduces the partial pressure of the volatile materials, allowing them to vaporize more freely.

BATCH OPERATION—In batch deodorization this injection steam also serves as a method of agitation of the oil. The undeodorized oil is placed in a vessel under vacuum and is heated by steam, Dowtherm vapor, or other means, through closed coils in the deodorizer, and when a certain minimum temperature is reached injection steam is introduced over the remaining period

of the deodorizing cycle to agitate and help distill out the volatile material. The finished deodorized oil is then cooled before allowing it to come in contact with air.

CONTINUOUS OPERATION—Continuous deodorization involves in general the same cycle of operations. In the first continuous installation the oil was partially heated before being subjected to the high vacuum, and although the stripping of volatile materials was satisfactorily accomplished and a good commercial product obtained, certain advantages were indicated in the process of placing the oil under high vacuum before and during the heating operation. This arrangement is incorporated in the latest installations.

Deodorization is accomplished in the continuous unit with only a portion of the injection steam required for batch operation because of the progressive and counter-current contacting of the oil with the stripping steam. The heated oil passes successively downward over a series of contacting trays. The stripping steam enters at the bottom tray, so that the pure deodorized oil is contacted only by the incoming pure injection steam, free of volatiles and fatty acids.

The contact tray consists of a series of nozzles through which the steam flows upward; each nozzle being fitted with a cap slotted at the lower edge, which directs the steam downward inside the cap and sideways through it, where it then bubbles through the relatively shallow layer of oil. These caps and trays are illustrated in Figure No. 2.

This method of steam and oil distribution gives very intimate contact and is much more efficient than steam blowing upward through a 7 or 8 foot depth of oil,—as is customary under batch operation.

The oil on the first or top tray flows across the tray

while it is being contacted by the up-flowing steam and then passes downward to the second lower tray, where this steam contacting operation is repeated. The same procedure is followed on downward through the successive trays installed in the deodorizing tower.

To illustrate the relative efficiency of this use of stripping steam:—

If there are ten (10) trays in the deodorizing tower, the up-flowing steam has ten times the chance to contact intimately the oil that it would have in the case of batch operation,—where it flows through only once. Therefore, the steam leaving the continuous deodorizer is more closely saturated with the volatile impurities that are being removed.

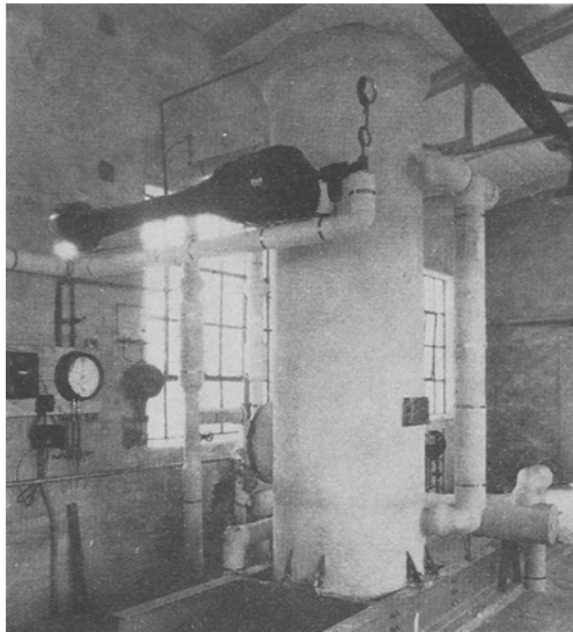
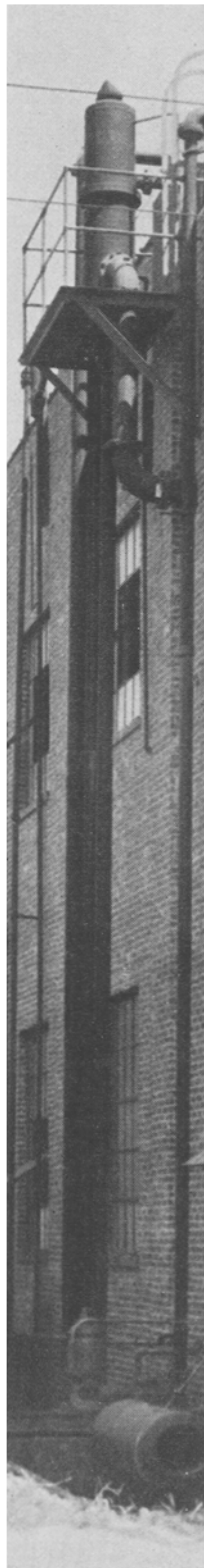


Figure 1. Cottonseed oil at the rate of 1200 lb. per hour has been processed in this deodorizer since 1938. The tower extends through two floors. High vacuum is maintained and objectionable constituents removed by the booster ejector with the assistance of stripping steam. These vapors are discharged through the wall to the barometric condenser outside the building at the left. On the upper floor, the Dowtherm steam superheater extends horizontally to the right from behind the tower. The Dowtherm oil heater is behind the tower at the left. On the lower floor are the Dowtherm vapor generator of 500,000 Btu. per hour capacity, with controls and gages and the pump for hot finished oil. The two large horizontal shells extending to the left constitute the oil-to-heat exchanger and oil cooler. The upper shell (insulated) is for preheating cold oil with finished oil, and the lower one (bare) for cooling the finished oil still further.

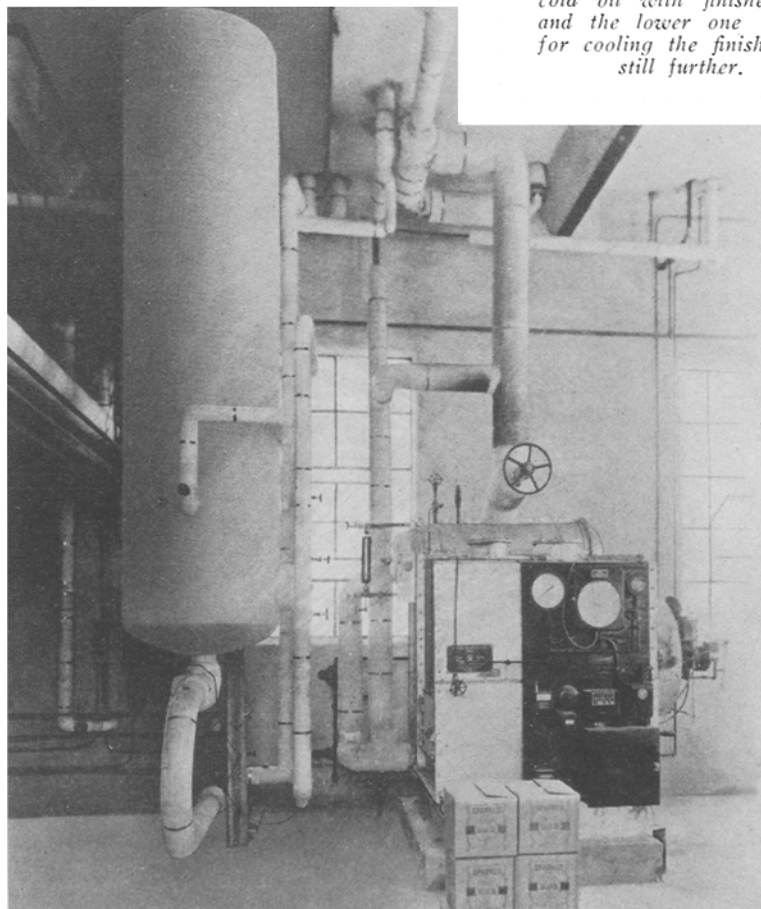


TABLE I
PROPERTIES OF SATURATED DOWTHERM VAPORS
DOWTHERM "A"

73.5% Diphenyl Oxide 26.5% Diphenyl

Temperature		Pressure		Heat Content tu./Lb.			Spec. Heat	Density Lb./Cu. Ft.	
° F.	° C.	Lb./sq. in. Abs.	Vacuum In. Hg.	Liquid	Latent	Total	Liquid	Liquid	Vapor
500	258	15	0.0	222.0	123	345	0.63	54.1	0.28
510	266	17	2.0	228.0	121	349	0.63	53.7	0.32
520	271	19	4.0	234.0	120	354	0.64	53.2	0.36
530	277	21	6.0	240.0	119	359	0.64	53.0	0.40
540	282	24	9.0	247.0	118	365	0.65	52.7	0.44
550	288	27	12	253.0	117	370	0.65	52.3	0.48
560	293	30	15	260.0	115	375	0.65	51.9	0.54
570	299	33	18	267.0	114	381	0.66	51.6	0.60
580	304	36	21	274.0	112	386	0.66	51.2	0.67
590	310	39	24	281.0	111	392	0.66	50.8	0.75
600	315	43	28	288.0	110	398	0.66	50.4	0.88
610	321	47	32	295.0	109	404	0.67	50.1	1.00
620	327	51	36	302.0	107	409	0.67	49.8	1.10
630	332	56	41	309.0	106	415	0.67	49.3	1.17
640	338	62	47	316.0	105	421	0.67	49.1	1.24
650	343	68	53	323.0	104	427	0.67	48.6	1.29
660	349	74	59	330.0	102	432	0.68	48.4	1.34
670	354	81	66	337.0	101	438	0.68	47.9	1.40
680	360	88	73	344.0	99	443	0.68	47.5	1.5
690	366	95	80	351.0	98	449	0.68	47.2	1.6
700	371	103	88	358.0	97	455	0.68	46.9	1.7
710	377	111	96	365.0	95	460	0.68	46.3	1.8
720	382	120	105	372.0	93	465	0.68	45.9	1.9
730	388	129	114	379.0	92	471	0.68	45.5	2.1
740	393	139	124	386.0	90	476	0.68	44.9	2.3
750	399	150	135	393.0	89	482	0.68	44.4	2.5

*Heat content is heat above freezing point, i.e., 53.6° F.

3. COMPARISON OF RELATIVE EFFECT OF TEMPERATURE, VACUUM AND INJECTION STEAM QUANTITY.

Even though maximum high vacuum of, say, .25" mercury absolute is obtained in a batch deodorizing vessel, only the oil on the upper surface is benefited directly by this high vacuum. At the bottom, say under a hydrostatic head of 8 ft. of oil, the effective vacuum would drop to 5" or 6" absolute. This explains the relatively high steam consumption of batch as compared with continuous deodorization, and also presents the possibility of hydrolysis of some of the glycerides at the base of the deodorizer; aside from the entrainment problem.

In the continuous cycle, with .25" mercury absolute at the top of the deodorizing tower, the relatively small pressure drop on each tray (approximately .06" of mercury per tray) on a basis of ten trays, results in a still relatively high vacuum at the bottom of the deodorizer

as an absolute pressure of .85" mercury as compared with an absolute pressure in the base of the usual batch deodorizer of approximately 6" to 7" mercury absolute. Figure No. 3 illustrates diagrammatically the absolute pressure differences at various points in both batch and continuous towers with the same top tower absolute pressure of .25" mercury.

4. INSTALLATION EXPERIENCES:

Since our discussion of this subject two years ago, three commercial installations have been added and a fourth is now being installed.

It is interesting to note that two of these installations—having capacities respectively of 5000 and 3000 lb. hourly—were made after a year of experimentation with a 1200 lb. per hour unit. Figure 4 is a diagram of a complete continuous system and Figure 5 shows installation of the 3000 lb. per hr. unit mentioned above.

The experimental unit was entirely constructed of plain or carbon steel. The two later commercial units,

as well as the fourth unit referred to as now being installed, used 18-8 stainless steel construction wherever oil is in contact with the equipment.

The use of stainless steel has been justified by the elimination of any possible formation of iron soaps, and elimination of possible contamination which might affect the quality of the finished oil, and many factors involved in the production of first-class products are insured by this type of construction.

Cleaning out of the continuous equipment of stainless steel construction is not required over long periods of time.

Record of operating requirements for one of the commercial units shows confirmation of the operating economies anticipated and can be illustrated by the following summary:—

Over the operating period observed 11,000,000 lb. of oil were deodorized by the continuous unit, this comprising production of several different types of edible products.

The operating requirements for this period are listed below and are based on unit costs as follows:—

Steam charged at 25c per 1000 lb.
 Electricity charged at 6 mills per K.W. hour.
 Water charged at 8c per 1000 gallons.
 The costs of these items, expressed in cents per 1000 lb. of oil processed, are as follows:—

Steam	8c
Fuel	8c
Water	2.4c
Electric power for pumping.....	1.3c

Total Operating Cost per 1000 lb. of oil...19.7c
 Operating Labor18.0c

Total Operating Cost, including labor, per 1000 lb. of oil.....37.7c

Comparison of this record with average costs on good batch deodorization will, we think, show that the continuous deodorization process will involve only approximately one-third (1/3) of the cost of batch pro-

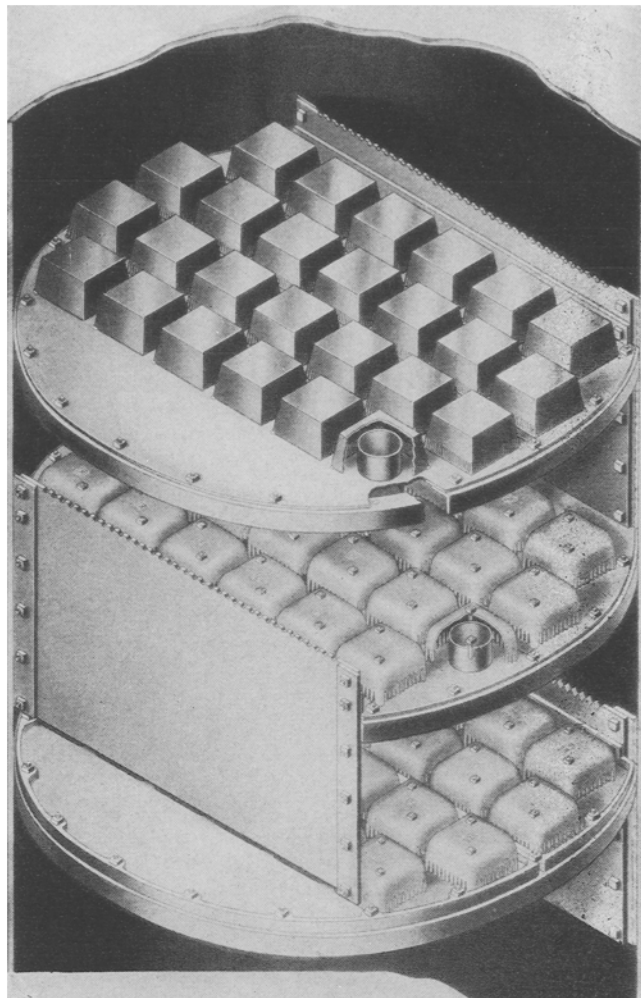


Fig. 2. Section through fractionating tower showing at the top stainless steel trays equipped with stainless steel nipples and bubble cups. The middle tray is of steel with cast iron caps and the lower tray is of cast iron with cast iron caps. Stainless steel trays and caps have proven most successful for treatment of vegetable and animal oils. Steam and noncondensable vapors pass through the nipples and are broken into small streams by the bubble caps as it passes in intimate contact through the liquid on the tray. Height of liquid on the tray is controlled by the dam over which the liquid flows onto the tray below. The oil is purified by stages as it passes over each tray on its way down through the tower.

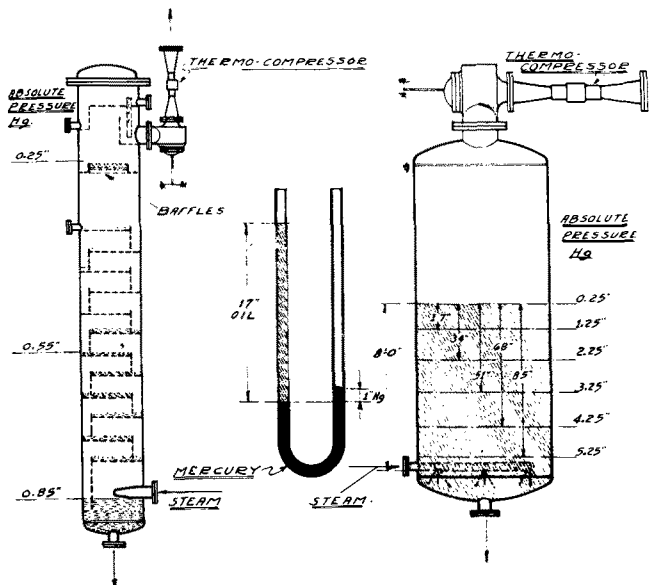


Fig. 3. Diagrammatic comparison of flash tower for a batch system and fractionating tower for a continuous deodorizing system. With the same top tower absolute vapor pressure of 0.25" mercury a 10 tray bubble tower would have a maximum vapor pressure at the bottom of 0.85" of mercury, whereas a batch system flash tank with 8 ft. of oil in the tank, would have a bottom vapor pressure of 5.25" mercury.

cessing—all to be based on the same unit costs of steam, water and fuel as referred to above.

In this connection it is worthy of mention that for every pound of injection steam used in either a batch or a continuous unit approximately 2 lbs. of steam will be required for the steam booster in maintaining the vacuum. Therefore, a reduction in injection steam to the continuous over the batch system correspondingly reflects in a multiple saving in total steam for the unit as a whole.

The same idea applies to the use of condensing water, —in that the water required for either batch or continuous operation would be in general proportionate to the total amount of steam coming to the condenser.

The saving in fuel for heating the oil is accomplished primarily in the continuous operation by the use of a heat exchanger in which the incoming raw oil to be deodorized is heated by the finished deodorized oil about to be discharged from the unit. The arrangement of such a heat exchanger is simple in a continuous operation as compared with a batch cycle and is therefore made thoroughly practicable in a continuous unit.

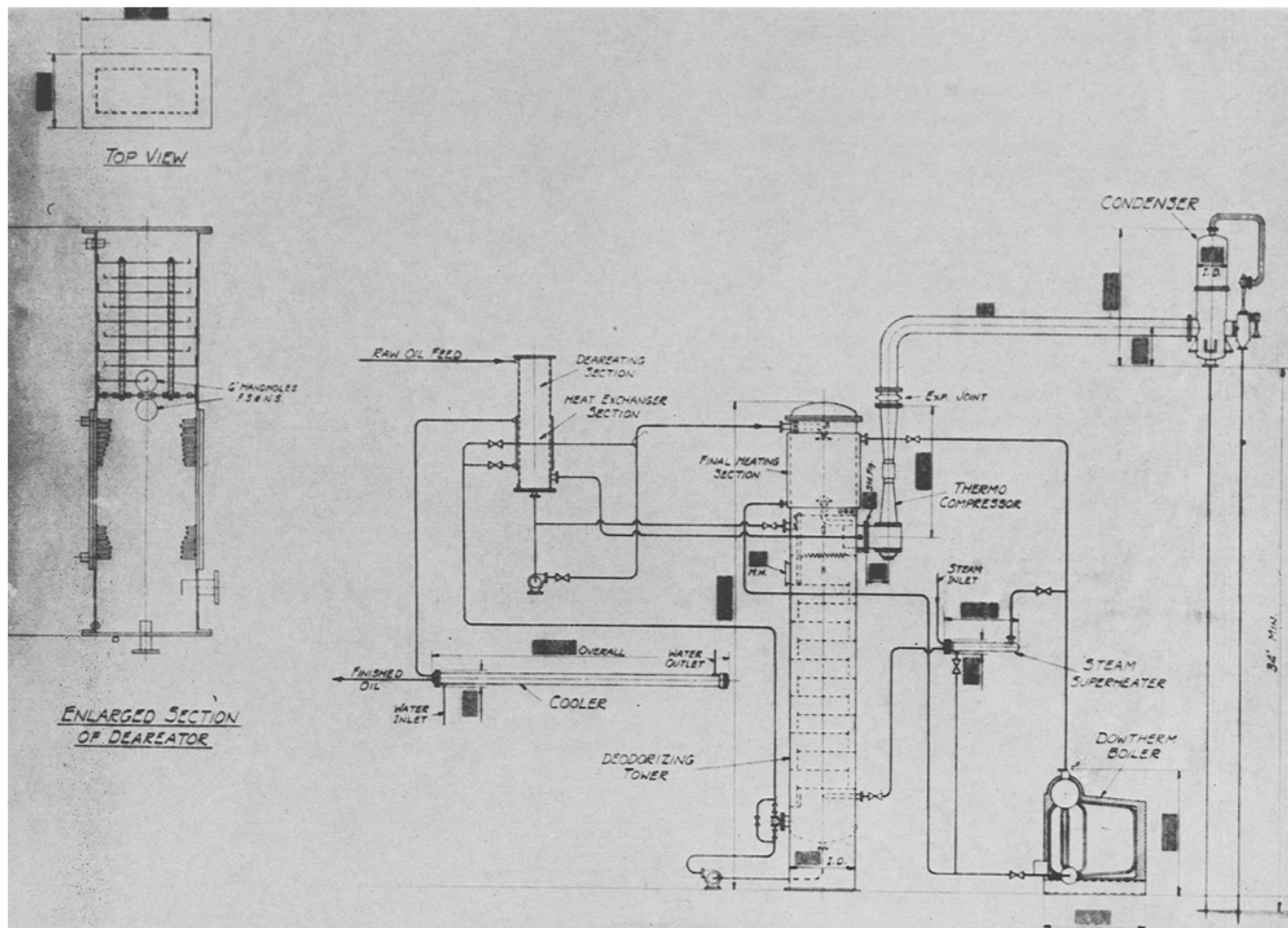


Fig. 4. Diagram of continuous deodorizing system with preliminary deaerating section for pretreating the oil before delivering it to the high vacuum bubble tower. Accurately controlled heating for the process is obtained by the use of Dowtherm vapor.

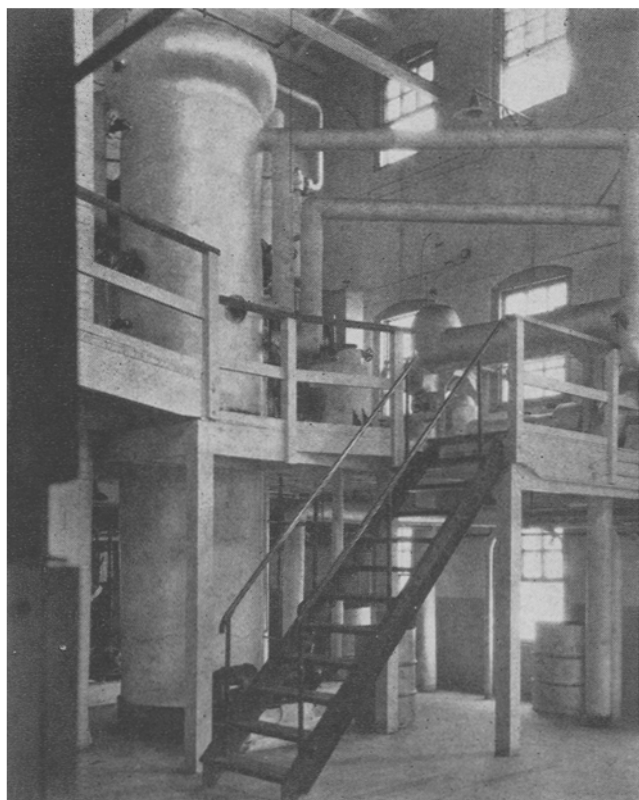


Fig. 5. Installation photograph of the top of the tower, heat exchanger, ejector and barometric condenser for a 3000 lb. per hour continuous deodorizing system.

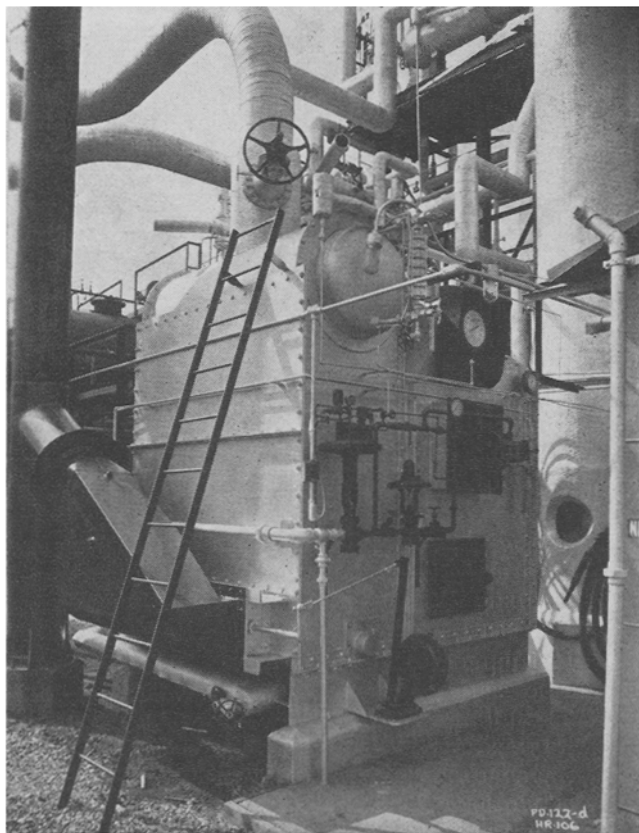


Fig. 6. Typical Dowtherm vapor generator as equipped with moisture proof steel casing for outdoor installation. This vapor generator has an output of 1,500,000 btu. per hour.

5. ENTRAINMENT LOSSES:

Early results from the first continuous deodorizer, with respect to negligible loss of glyceride, have been confirmed in the later installations. This considerably lower loss in continuous versus batch operation is not due to any magic but can be explained by the functioning of the two types of operation:—

First, if a batch unit as well as the continuous were fitted with the same efficient type of entrainment eliminators, there should still be considerably less loss in the case of the continuous deodorizer, by virtue of the greatly reduced quantity of injection steam blowing through the oil,—since entrainment from this source would be in general comparable with the amount of steam passing through the oil.

Secondly, there is no loss due to hydrolysis of glycerides in a continuous unit, which might result in the formation of the more volatile constituents such as fatty acids and glycerine,—as may occur in the relatively deep batch of oil in the batch process.

6. METHODS OF HEATING:

The continuous deodorization units so far installed

have incorporated the use of Dowtherm vapor generators for producing Dowtherm vapor for heating of the oil.

Although in some cases there might be sufficient steam pressure available to give adequate temperature,—Dowtherm (having a boiling point of 500° at atmospheric pressure) gives a high temperature heating medium usable at relatively low pressures; resulting in corresponding ease of control, elimination of high pressure piping, gauges, valves and fittings,—and has proved to be eminently satisfactory. This is proved not only by its success in continuous deodorization units, but by its general acceptance by the vegetable oil industry as applied to batch deodorization as well. Table No. 1 gives the properties of Dowtherm liquid and vapor and Figure No. 6 shows an outdoor installation of a Dowtherm vapor generator with a capacity of 1,500,000 Btu. per hour.

The fuel efficiency of Dowtherm vapor generators is relatively high, so that the cost of production of Dowtherm heat is comparable with that of high pressure steam produced by large steam generators.

The Evaluation of Oil Meal Color By the Methods of Photometry

By EGBERT FREYER

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THE present official method for grading meal samples as "prime" or "off" in color consists in comparing the meal with a strip of colored paper, revolving both at high speed to eliminate texture differences. "The color of the meal must be equal to or lighter than the standard to establish grade." For the grading to be reasonably precise this procedure must assume that all meals and the standard have the same hue, and that different specimens of a given standard all have the same color dimensions. Neither of these conditions is fulfilled. During an abnormal growing season in certain localities a large proportion of meal samples may have hues appreciably different from that of the standard, and even during normal years chemists may receive abnormal samples which it is impossible to match visually against the standard. In such instances it is apparent that, in borderline cases, the chemist's decision is largely arbitrary. Concerning the constancy and uniformity of the Munsell standards, the writer has no definite information—only what is implied by the fact that the one standard strip in his possession differs by about 10% in the brightness of the two ends. Now spectrophotometry, the science of exact color evaluation, provides methods for dealing with this situation as precisely as it affords complete color definition of transparent materials, of which an outstanding example is represented by the spectrophotometric color analyses of vegetable oils made at the Bureau of Standards.¹ But just as we have considered such methods to be impractical and too expensive for everyday commercial use in

the case of oils, they may likewise be dismissed with reference to meal color.

After a few preliminary trials in matching meal colors against different comparison standards, in which a prime condition was control of brightness of the two fields, the writer became convinced that grading the *brightness* of meal could be easily done without elaborate apparatus. The writer here avoids the use of the words, color or hue. If the industry requires a method which indicates differences of dominant wave length reflected, or in the subjective sense, of hue,—then the result can only be attained by expensive apparatus and an exacting technique; and any attempt to compromise will only result in such inadequacy and confusion as occasioned the formation of the present Meal Color Committee.

Aside from the desirability of forgetting hue differences in grading for the sake of using a simple and accurate brightness tester is the consideration that newer processing methods are likely to result in the production (from sound, prime seed) of meal with a somewhat different hue—such as would, perhaps unjustly, be called "off" under the present method, but which might at the same time be even *brighter* than meal made by old conventional methods. In fact, accounts have reached the writer which indicate this very situation to be arising, if not in a commercial sense, at least in some Company laboratories.

However the Society and the Industry may decide on this point, the writer's present efforts have been con-