Deutsche Hydrierwerke A.-G.—*Ger. 692,927, Cl. 12p.*

E. I. du Pont de Nemours & Co.—*U. S. 2,268,395.*

Emulsol Corp.—*U. S. 2,238,901, 2,238,927-9, 2,-239,706, 2,239,720, 2,239,997, 2,245,593, 2,248,089.*

J. R. Geigy A.-G.—*Brit. 513,663; Fr. 848,003; Swiss 206,594, 206,718, 207,299-303; U. S. 2,211,280. 2,214,971, 2,221,914, 2,229,803.*

General Aniline & Film Corp.—*U. S. 2,211,001, 2,218,939, 2,222,208, 2,230,591, 2,231,502, 2,267,725.*

Imp. Chem. Ind. Ltd.—*U. S. 2,237,296.*

I. G. Farbenind. A.-G.—*Fr. 847,599, 849,018-20; Ger. 697,170 Cl. 8k, 697,730 Cl. 12o; U. S. 2,210,442, 2,215,367, 2,251,892.*

Katz—*U. S. 2,216,618.*

Neu—*Ger. 688,083 Cl. 12p.*

No. Am. Rayon Corp.—*U. S. 2,242,223-4.*

N. V. de Bataafsche Petroleum Maatschappij—*Fr. 849,399.*

O'Connor—*U. S. 2,244,721.*

Petrolite Corp. Ltd.—*U. S. 2,228,985-9.*

Pollak—*U. S. 2,257,481.*

Purdue Research Foundation—*U. S. 2,247,106.*

Resinous Products & Chem. Co.—*U. S. 2,210,831, 2,260,967.*

Reichold Chemicals Ind.—*U. S. 2,215,038.*

Röhm & Haas Co.—*U. S. 2,229,024.*

Searle & Co.—*U. S. 2,252,863.*

Shell Development Co.—*U. S. 2,247,266.*

Soc. pour l'ind. chim. a Bale—*Brit. 527,008, 527,-012; Fr. 849,147; Swiss 208,530 Cl. 12o, 208,534 Cl. 36p, 209,972 Cl. 36p; U. S. 2,226,057.*

Squibb & Sons—*U. S. 2,251,946.*

A miscellaneous group of detergents included various organic esters and phosphated, borated and some halogenated compounds:

Chemische Fabrik Joh. A. Benckiser—*U. S. 2,-252,479.*

Colgate-Palmolive-Peet Co.—*Brit. 514,053.*

Deutsche Hydrierwerke A.-G.—*Ger. 694,944 Cl. 12o.*

Doelling—*U. S. 2,255,916.*

E. I. du Pont de Nemours & Co.—*U. S. 2,254,124.*

Emulsol Corp.—*U. S. 2,236,516.*

J. R. Geigy A.-G.—*U. S. 2,212,224.*

General Aniline & Film Corp.—*U. S. 2,217,846, 2,228.929.*

I. G. Farbenind. A.-G.— Ger. 693,028 Cl. 8i, *696,317 Cl. 12o.*

Röhm & Haas Co.—*U. S. 2,249,111.*

Soc. pour l'ind. chim. a Bale—*Swiss 207,645.*

Glycerin Recovery—The Soap Kettle

WM. J. GOVAN, JR.
Pacific Soap Co., Ltd., San Diego, Calif.

Introduction

In these times it is of great importance that the greatest feasible amount of glycerin be recovered from the fats in the soap kettle.

According to the bulletin of the U. S. Department of Commerce "Animal and Vegetable Fats and Oils" for the years 1936-1940 inclusive, the amount of fats consumed in soap manufacture was, roughly, 5,745,-000,000 pounds. The amount of 80 per cent crude produced for the same period of years was 865 000,-000 pounds, which is equivalent to 692,000,000 pounds of 100 per cent glycerol. About 23 per cent of the fats consumed were in the cocoanut oil group which contains a greater proportion of glycerin than the usual animal and vegetable fats. On the assumption that the fats consumed in the soap kettle averaged 94 per cent glycerides, the theoretically available glycerol was about 864,000,000 pounds (as 100 per cent glycerol). The efficiency of recovery of the soap industry was 80 per cent of the theoretical. It is true that glycerin was completely lost in the so-called cold-made soaps and in soft and liquid soaps. This loss was probably more than offset by the high efficiency attained in the large-scale fat-splitting operations. Therefore, it is probably a fair assumption that the average countrywide recovery of glycerin, using the soap-boiling method of manufacture, is in the order of 80 per cent or less. A rise from 80 to 85 per cent in recovery would be equivalent to a yearly increase of 9,000,000 pounds of C. P. and dynamite grade glycerin. This is a mark for the soap industry to shoot at. This paper will show that an increase to a 90 per cent recovery or better is entirely within the range of the individual soap factory with no more than the usual existing kettle facilities.

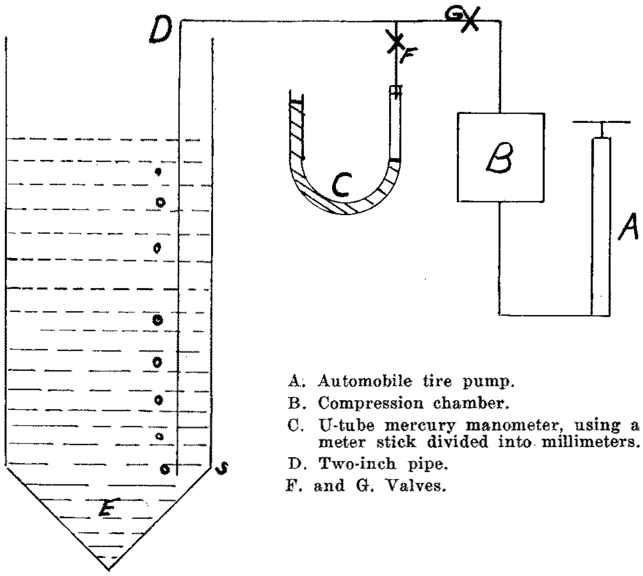
Preliminary

It has been established that for practical purposes, glycerol is of equal concentration (1, 2) both in the lye and in the aqueous portion of the curd. To put it more simply, if the watery portion of the curd contains 5 per cent glycerol, it follows then that if the weight of the lye and the weight of the aqueous portion of the curd is known, it can be estimated what percentage of free glycerol has been removed from the kettle by the lye. Conversely, by regulating the amount of lye relative to the size of the kettle, a definite percentage of glycerol can be removed from the kettle. Extending the latter idea to its end, it is evident that a definite kettle recovery of glycerin can be obtained by being able to regulate the weight of the "washes" relative to the weight of the curds. (The curd under ordinary kettle operation will hold 35-38 per cent of its weight in "entangled lye.")

The Weighing Apparatus

The apparatus used for weighing the washes is fully described (3) by J. H. Wigner.

The following sketch and description is the author's home-made adaptation of the apparatus:



A. Automobile tire pump.
B. Compression chamber.
C. U-tube mercury manometer, using a meter stick divided into millimeters.
D. Two-inch pipe.
F. and G. Valves.

Operation of Apparatus

Valve "F" is closed, "G" is left open. Air is pumped into the system until bubbles break through the surface of the kettle. Valve "G" is closed, Valve "F" is opened and the difference in millimeters between the two levels of mercury in the manometer is noted down.

Millimeters X "kettle factor" will give the total weight of the kettle contents above the opening "S" of the pipe line "D" in the bottom of the kettle. The weight of the kettle contents in the cone below the opening "S" is to be regarded as a constant "kettle tare" and may be obtained directly or computed. The "kettle factor" itself may be computed from the horizontal cross section of the kettle or may be calibrated directly by noting the manometric differential against a known weight of material pumped into the kettle. (The factor for a kettle fifteen [15] feet in diameter is 490 pounds for each millimeter head of mercury. The tare on the cone section below the pipe opening "S" for a cone 2½ feet deep is 9,000 pounds for lye and 6-7,000 pounds for kettle soap.) The accuracy of these weighings is independent of the condition of the material in the kettle. It can be nonhomogeneous and at any temperature as long as fluidity is maintained.

How the Apparatus Is Used

In the operation of the kettle, the total contents are built up to a specific weight. After the wash is run out, the curd is also weighed. The difference between the total weight of the kettle and the weight of the curd gives the weight of the lye withdrawn. The aqueous portion of the curd is estimated by deducting the known amount of dry charge from the curd. The percentage removal of free glycerin from the kettle is the ratio of wash to the total aqueous medium in the kettle.

Another advantage obtained from this apparatus is that the "efficiency of the curd" may be obtained. If a kettle is "oversalted," or is not allowed sufficient settling time, then the curd will retain more entangled lye than it should. This will adversely affect the percentage glycerin removal. Under correct operation, curds should not contain more than 35-36 per cent entangled lye. Thus by keeping a record, proper salt concentration in the lye and proper settling time may be ascertained to insure an efficient curd.

Details of the Kettle Plan Evolved from the Foregoing Principles

EXCERPT FROM COUNTERCURRENT WASH KETTLE PLAN

Day	Kettle A	Kettle B	Kettle C
1	Change No. 1 Lye to glycerin department		Change No. 3 Lye to "A"
2	Change No. 2 Lye to "B"		Settle
3	Change No. 3 Lye to "B"	Change No. 1 Lye to glycerin department	
4	Settle	Change No. 2 Lye to "C"	
5	Neat pumped	Change No. 3 Lye to "C"	Change No. 1 Lye to glycerin department
6		Settle	Change No. 2 Lye to "A"
7	Change No. 1 Lye to glycerin department		Change No. 3 Lye to "A"

The first change is carried to nearly complete saponification (usually 95 per cent) with fresh caustic and the size of the wash is regulated to an amount which will remove 70 per cent of the residual free glycerin from the kettle.

In the second change, saponification is completed by "closing" the curd with water and boiling it in that state with an excess of fresh caustic for an hour. This wash is also built up to remove 70 per cent of the residual free glycerin.

In the third change this wash, unlike the first two washes, is built up entirely of fresh brine or caustic and added water. The removal of residual glycerin from this change is 50 per cent.

In the settle, the niger retains about one-third of the small amount of glycerin left at this stage.

Discussion

Operating a kettle under this flow plan with a charge of tallow yielding, for example, 10 per cent of its weight in glycerol, these results will be obtained:

- Net kettle recovery of glycerol—94 per cent of the theoretical
- Ratio of pounds of spent lye to pounds of net kettle fat 1.35/1.00
- Concentration of glycerol in spent lye, weight-to-weight, 7 per cent
- Production capacity per six-day kettle cycle 24.5 pounds neat soap/cubic foot kettle space.

The above results are now being achieved in this plant with the 70-70-50 per cent ratio washes. The experience of the first four months' operation under this plan using 65-65-45 per cent ratio washes gave an inventoried glycerol recovery well over 85 per cent.

The formula below will give the percentage of recovery in the range of 80-95 per cent with fairly accurate results on a countercurrent, three-wash kettle plan in which the new stock is about 95 per cent saponified in the first change.

This formula may be used to estimate the recovery on a given kettle or it may be used in setting up a kettle plan with the purpose of achieving a definite recovery.

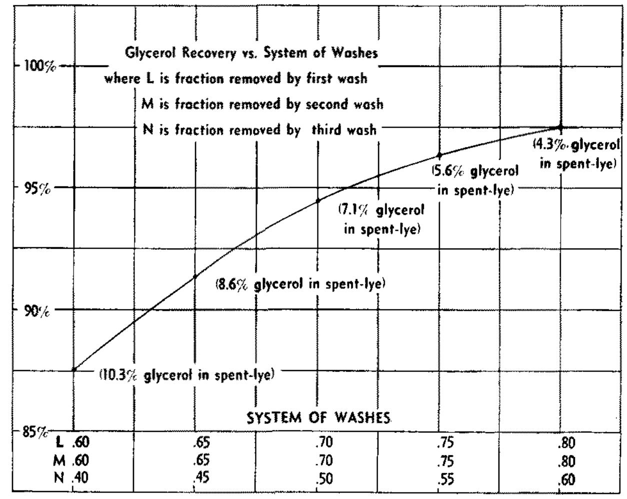
Summary

A kettle plan has been worked out which in actual practice yields a well-cleansed neat soap, with ample production, and with a predictable high recovery of glycerin.

A formula has been set up which in the hands of a chemical engineer will enable him to recover the highest practicable yield of glycerin.

Acknowledgment

The author wishes to thank Eugene Hamry, chief soapmaker, for his cooperation. Without his skillful, intelligent craftsmanship this work would have been much more difficult.



BIBLIOGRAPHY

(1) Wigner, J. H., Soap Manufacture, Page 116, Chemical Publishing Co., 1940.

(2) Ferguson, R. H., Oil and Soap, Vol. XIV, No. 5.

(3) Wigner, J. H., Soap Manufacture, Page 56, Chemical Publishing Co., 1940.

$$\% \text{ Recovery} = 100L \left[(.950+A) + \frac{(1.000+A)M - (.950+A)LM + \dfrac{(MN)(1-M)(1+A-.950L-LA)}{(1-N+MN)(1-M+ML)}}{(1-M+LM)} \right]$$

where "L" is the ratio of wash to total aqueous content of kettle on the first change
"M" is the ratio on the second change
"N" is the ratio on the third change
"A" is an element the value of which varies according to the system of washes used.

System	*Value of "A"*
.60-.60-.40	.060
.65-.65-.45	.043
.70-.70-.50	.026
.75-.75-.55	.016
.80-.80-.60	.012

Abstracts

Oils and Fats

Edited by
M. M. PISKUR and SARAH HICKS

Coconut oil. *Soap 18*, No. 3, 21-4, 67 (1942).

A review of the role of lipids in the manufacture of leather. Robt. M. Koppenhoefer. *J. Am. Leather Chem. Assoc. 37*, 118-31 (1942).

Fats for candy, meeting the coconut oil shortage. J. A. O'Malley and C. E. Price. *Manuf. Confectioner 22*, No. 3, 13-15 (1942).

Studies on the chemistry of the fatty acids. IX. A spectroscopic study of methyl arachidonate purified by crystallization and distillation and its alkali isomerization product. D. T. Mowry, W. R. Brode, and J. B. Brown. *J. Biol. Chem. 142*, 671-8 (1942). A critical study of the method of isolating methyl arachidonate by crystallization and distillation has been made and the product thus obtained compared with that prepared by reduction of the octabromide. The absorption spectra of these samples show even better than the diene no. and molecular refraction that these samples are relatively free from conjugated unsaturation. A correction has been recommended for the empirical factor in the equation for the estimation of methyl arachidonate by the polybromide no. The kinetics of the alkali isomerization of arachidonic acid have been followed spectroscopically and the similarity to the behavior of the 1, 4, 7-triene system present in linolenic acid noted. The alkali isomerization product of arachidonic acid has been described.

Adrenalectomy and the absorption of different fats. R. H. Barnes, I. I. Rusoff, and Geo. O. Burr. *Proc. Soc. Exptl. Biol. Med. 49*, 84-7 (1942). The rate of absorption of corn oil, olive oil, hydrogenated vegetable oil, and mutton tallow is not significantly changed from the normal by adrenalectomy. The absorption of hydrogenated vegetable oil fed as an emulsion with skim milk is significantly decreased following this operative procedure. Evidence for certain changes in fat absorbing mechanisms that are brought about by removal of the adrenals is presented, and it is pointed out that these changes need not result in an altered rate of absorption.