The Countercurrent Wash System in Soap Making*

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THE most widely used commercial method of soap manufacture in the United States is the "full boiled" method. It may be safely estimated that at least three quarters of the hard soap manufactured in the United States is made by the full-boiled method utilizing neutral fats as a base. Other methods of commercial importance, such as the "semiboiled," "cold-made" and "continuous," are used to some extent but so far have limited application.

Boiling is conducted in large cylindrical or square kettles with capacities ranging from a few thousand pounds of stock to several hundred thousand pounds. Kettles are generally made of steel. Some are made with the various corrosion-resistant, clad steels in whole or in part to avoid product contamination. Boiling is carried on by means of open steam, although closed steam coils are sometimes used. Processing materials are introduced at the top of the kettle. Finished neat soap is withdrawn from the kettles by means of a swing or skimmer pipe. Lyes and niger are drawn off at the bottom of the kettle.

Glycerine is a valuable chemical produced by the saponification of fats in soap boiling. Therefore, the efficiency of glycerine recovery, both from the soap kettle and also from the treatment and evaporation of spent soap lyes, is an important factor in the profitable operation of the soap plant. This paper will include a discussion of recovery of glycerine from the soap kettle. The treatment and evaporation of spent soap lyes have been discussed in other papers (2, 5).

Kettle Operations

Three basic operations take place during the course of soap boiling, viz., saponification, washing, and fitting.

Saponification. Fats charged to the kettle are saponified with caustic soda. The chemical reaction results in the formation of glycerol and sodium salts of fatty acids or soap.

Washing. The function of the wash is to remove coloring matter, impurities and glycerol from the kettle of soap. The method of performing a wash is to "grain" the kettle of soap by the addition of salt, caustic soda, or a combination of the two. A kettle when properly grained will separate into two layers. The upper layer is called the curd, and under good practice consists of from 60% to 64% anhydrous soap; the balance being water containing a small amount of electrolyte. The curd is not uniform throughout.

The lower layer is soap lye consisting of an aqueous solution of salt, caustic, or both. This is drawn off preparatory to a further change.

Fitting. The third basic operation is the fit. The purpose of the fit is to produce a kettle soap of a texture suitable for further processing into finished products. After the soap lye has been removed from the final wash, leaving only a curd, fresh water is added to the curd under constant boiling to dilute its electrolyte content. When the fit is carried out properly, the kettle will settle into two layers. The upper layer, roughly amounting to three quarters of the total kettle, is neat kettle soap. This layer of relatively pure soap is fairly homogeneous throughout and may vary in anhydrous soap content from kettle to kettle in the range of 65% to 69%. The balance is mainly water, glycerol, and a small amount of electrolyte.

The lower layer is called the niger. It is a nonhomogeneous mass and generally averages less than 40% anhydrous soap. A large portion of the impurities and coloring matter of the kettle settle into the niger, giving it its dark color. The niger is reused in a succeeding kettle.

Distribution of Glycerol in the Kettle

It is well established (1, 4) that glycerol is distributed uniformly throughout the aqueous portion of the kettle. For example, if the aqueous part of the soap lye layer, excluding dissolved solids, contains 5 per cent glycerol, then the corresponding aqueous part of the soap curd will also contain 5 per cent of glycerol.

Furthermore, in ordinary kettle practice, 100 lb. of anhydrous soap in the curd will hold approximately 61 lb. water and glycerol, and 100 lb. soap lye will hold approximately 90 lb. water and glycerol. These figures will vary somewhat over a limited range according to composition of soap base and existing kettle practices.

Therefore, by regulating the size of the soap lye wash with respect to the curd, it is possible to remove a definite percentage of free glycerol from the kettle. It is only a further step to set up systems of washes with regard to size and number for the purpose of effecting a definite percentage recovery of the glycerol available in the fats charged to the kettle.

Measurement of Kettle Contents

In order to regulate the size of washes in the soap kettle, means must be provided for measuring the contents of the kettle during boiling. This may be accomplished by means of a back pressure (4) liquid level gauge, either the manometer or dial type. These gauges are obtainable from instrument and gauge manufacturers, or may be made locally. It is desirable that the air column pipe which goes to the bottom of the kettle be a two-inch pipe because in smaller sizes there is a tendency for soap to "set up" inside the pipe and block the flow of air, thereby making a correct gauge reading impossible.

The accuracy of this method of weighing the contents of a kettle is generally ± 2.0%, which affords adequate control over operations.

System of Washes

There are three general systems of washes used in the full boiled method of soap making. The main

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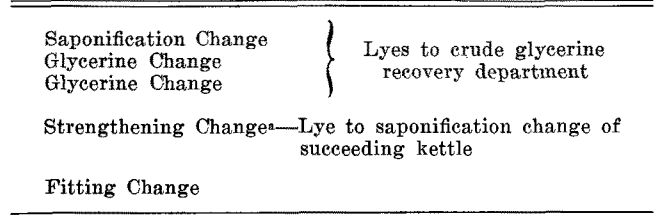


[a] Optional.

FIG. 1. Individual wash system.

purposes of washes are to cleanse impurities and coloring matter from the soap and to recover glycerine which the fats and oils yield on saponification.

Individual Wash System. The earliest system of washes (see Figure 1) is one in which several successive fresh neutral washes are made on the same kettle for glycerine recovery. Saponification and fitting operations are carried out in the customary manner.

This system of washes is still much used where the number of kettles is limited and various grades of stock must be handled without the possibility of contaminating good soap stock by countercurrent washes from poorer soap stock.

The individual system of washes has one major drawback, viz., production of a relatively large amount of spent soap lye compared with the amount produced in countercurrent systems. Consequently, in the individual wash system, labor, material, and steam costs for evaporating spent lye to crude glycerine are oftentimes double those in the countercurrent system (see Figure 5). This places the former system at an obvious economic disadvantage.

Semi-Countercurrent System. A second system of washes, semi-countercurrent (see Figure 2), is a compromise between the individual system just described and a straight countercurrent system. In this system several fresh washes of varying caustic soda strength are drawn off the same kettle into storage tanks. These washes are later used in the saponification or "killing" change in succeeding kettles where they are "enriched" by picking up more glycerine and where their caustic soda strength is spent in saponifying fresh fats and oils.

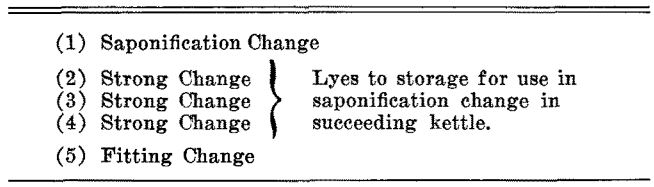


FIG. 2. Semi-countercurrent system.

The semi-countercurrent system has the same advantage as the individual wash system for soap plants having limited kettle facilities. A variety of soap stock can be processed with little possibility of the contamination of good stock through interchange of lyes.

It has the advantage over the individual wash system of reducing the amount of spent soap lye produced by about 25 percent with a proportionate reduction of operating costs in the crude glycerine plant.

Here again there are serious disadvantages in this system compared with the straight countercurrent system. Over one and one-half times the amount of spent soap lye is produced in the semi-countercurrent system as in the countercurrent system. This means that labor, material, and steam costs of producing crude glycerine are about one and one-half times those in the countercurrent system.

A second disadvantage is in the extra amount of time, labor, and expense consumed in running partly spent lyes from the kettle to storage tanks and then back to a succeeding kettle for the saponification change. These partly spent lyes may cool off on storage and in that condition the reaction between their caustic soda content and the fat charge is slow and uncertain.

Countercurrent System. The third system of washes is the countercurrent system (see Figures 3 and 4). In this system, a wash is pumped directly from one kettle to a succeeding kettle which is in an earlier stage of the process. The same wash will pass through a number of kettles until its countercycle is completed and it is transferred to the glycerine department as spent lye.

In larger soap plants where the countercurrent system is used, kettles receive ten or more washes on a twenty-four hour a day kettle schedule with short settles between washes. The recovery of glycerol from the saponification of the fats is approximately 95% of that theoretically available. This recovery is accomplished with a ratio of less than one pound of spent lye produced per pound of fresh fat or oil charged to the kettles.

In the medium or small soap plant, where it is not feasible to conduct a twenty-four hour a day schedule on the soap kettles, the countercurrent system is applicable, using about three large washes per kettle and overnight settles between washes.

Typical Operation of Three-Wash Countercurrent System

Proper operation of the three-wash countercurrent system requires units of four kettles of equal capacity. Each unit is independent of other units and handles one grade of soap stock in its four kettles.

The changes in each unit are so staggered that washes from kettles in a more advanced stage are pumped directly to kettles in a less advanced stage. For a three-wash schedule, the cycle of each kettle from the initial charge until the kettle is ready to be charged again is eight days. One kettle of the unit is finished every second day, thus providing a steady, continuous supply of neat soap. A unit of four kettles will produce at the rate of approximately 3.5 pounds of neat soap per day per cubic foot of kettle space.

Each kettle is charged with a weight of fresh fat equal to approximately three-tenths the gross capacity of the kettle calculated as water.

When the operation is started for the first time on empty kettles, the fat charge is increased above the normal charge by about 15% to allow for soap stock equivalent to that in a normal niger. The kettles are not put into operation simultaneously, but one is started on every second day until all are in operation. Washes are made up of fresh caustic lye, salt, and water, until washes from other kettles in the unit become available.

Figure 3 shows the schedule for the four-kettle unit in which the three-wash countercurrent system is used. The pattern of the eight-day cycle as shown

is repeated continuously during the operation of the plant.

For the purpose of illustration, the flow sheet in Figure 3 is based on a kettle charge of 50,000 lb. of fats and oils which yield, on saponification with caustic soda, 51,500 lb. anhydrous soap and 5,000 lb. glycerol.

If the kettle charge is greater or less than 50,000 lb., the kettle compositions shown below will vary in proportion to the charge.

The composition of the kettles on each change is approximately as follows:

First Change

Curd:		
Anhydrous Soap	46,400 lb.	
Unsaponified Fat	5,000 lb.	
NaCl and other dissolved solids	900 lb.	
Glycerol	2,360 lb.	
Water	25,240 lb.	
Total		79,900 lb.
Lye:		
NaCl and other dissolved solids	6,200 lb.	
Glycerol	4,730 lb.	
Water	50,470 lb.	
Total		61,400 lb.

Second Change

Curd:		
Anhydrous Soap	51,500 lb.	
NaCl, NaOH, and other dissolved solids	1,000 lb.	
Glycerol	1,055 lb.	
Water	29,445 lb.	
Total		83,000 lb.
Lye:		
NaCl, NaOH, and other dissolved solids	8,300 lb.	
Glycerol	2,590 lb.	
Water	72,310 lb.	
Total		83,200 lb.

Third Change

Curd:		
Anhydrous Soap	60,600 lb.	
NaCl, NaOH, and other dissolved solids	1,200 lb.	
Glycerol	405 lb.	
Water	35,495 lb.	
Total		97,700 lb.
Lye:		
NaCl, NaOH, and other dissolved solids	7,700 lb.	
Glycerol	785 lb.	
Water	68,715 lb.	
Total		77,200 lb.

Fourth Change

Neat:		
Anhydrous Soap	51,500 lb.	
NaCl, NaOH, and other dissolved solids	300 lb.	
Glycerol	270 lb.	
Water	26,830 lb.	
Total		78,900 lb.
Niger:		
Anhydrous Soap	9,100 lb.	
NaCl, NaOH, and other dissolved solids	900 lb.	
Glycerol	130 lb.	
Water	12,970 lb.	
Total		23,100 lb.

Changes for Kettle A only are described below. The same changes on the other kettles are performed in the same manner.

First Change. Change No. 1, sometimes called the "saponification" or "killing" change, is given to Kettle A. The niger from the previous boil in this kettle has been pumped over to storage and the new boil is begun on an empty kettle. Vigorous boiling is carried out with open steam while the fat stock and the soap lye from Change No. 2 of Kettle D are pumped into the kettle. The caustic strength of the lye is "spent" or used up in saponifying the fat charge. Additional fresh caustic lye is needed to carry the saponification further. Virtually complete saponification can be accomplished at this stage by skillful operation and with sufficient time. However, it is practical to saponify only 85% to 90% of the fresh charge because the extra time required for complete saponification is not justified by any appreciable added benefit.

It is important that a proper balance of electrolyte be maintained in the kettle during the saponification. Too little electrolyte causes "bunching," resulting in a highly viscous mass of soap in which agitation and intermixing become nearly impossible. Too much electrolyte on the other hand grains out the soap and retards the progress of saponification. It is well to keep the soap in a semi-pasty or closed condition throughout this operation.

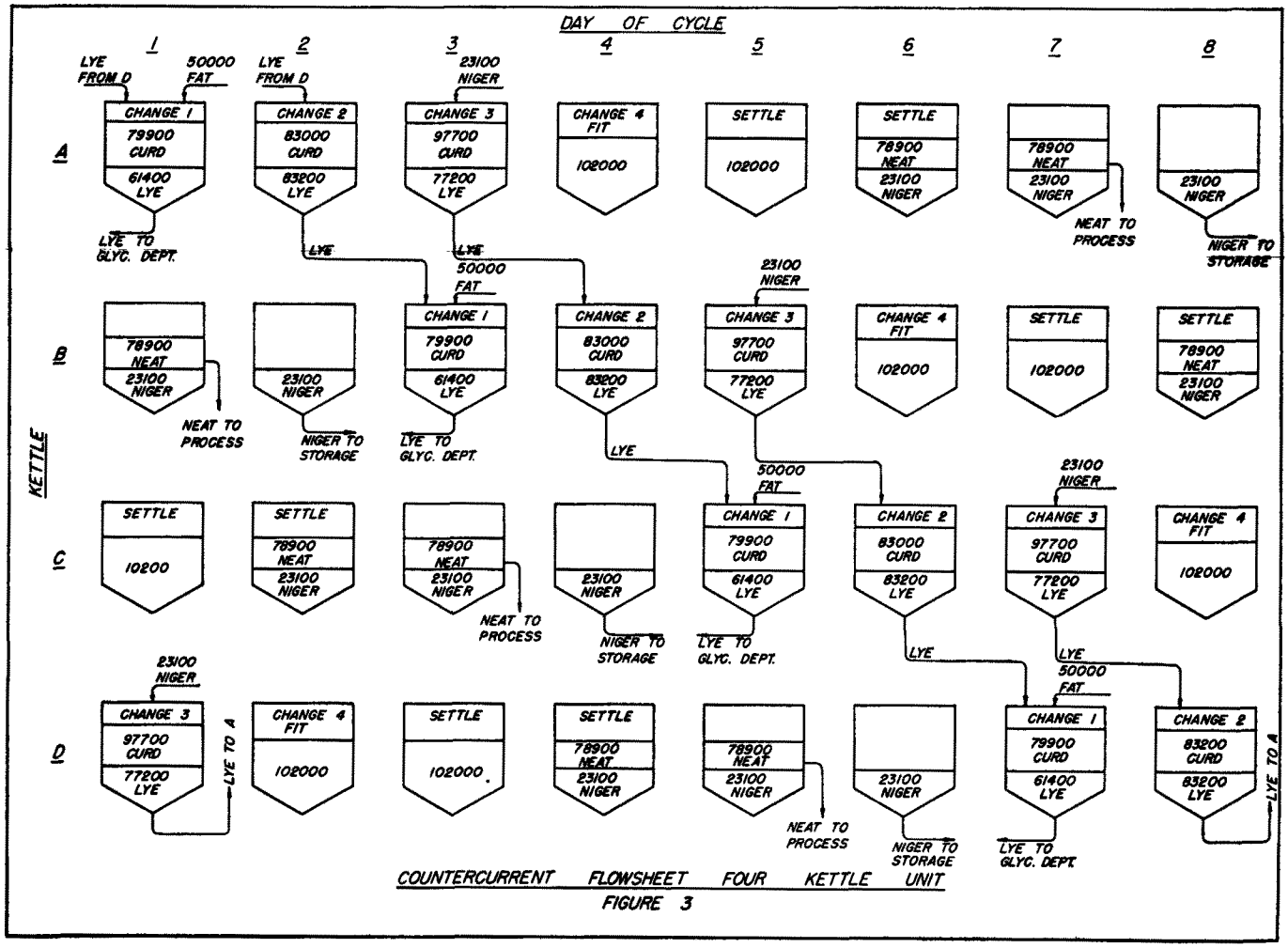
If saponification is carried out properly, it should take no more than two or three hours to saponify upwards of 50,000 lb. fat to the extent of 90% of the fat charge.

Toward the end of the saponification phase of the first change, the kettle begins to take up additions of fresh caustic lye more and more slowly. This is an indication that saponification has gone as far is practical. Before proceeding to grain out the soap with salt, the kettle should be boiled thoroughly until there is no free caustic left. A suitable indicator for detecting free caustic is thymolphthalein solution or test papers. If no color shows on using the indicator or if only a faint blue color appears, the kettle can be considered neutral and ready for the graining out operation.

When the kettle is neutral, it is grained out with dry salt. Concentrated brine is sometimes used but it increases the amount of spent lye unduly. Great care must be exercised in obtaining the right concentration of salt in the lye. Insufficient salt results in a spent lye containing a large amount of dissolved soap. Not only is this soap wasted during the treatment of spent lyes, but its presence makes satisfactory treatment difficult.

Too high a salt concentration in the lye causes a condition known as "oversalting." The curd has a "hard grain" and tends to hold a greater percentage of water than is normal. This reduces the efficiency of the glycerine recovery since a greater portion of glycerol is retained in the curd and a lesser portion of glycerol is removed with the spent lye.

There are several methods of determining when a "grained" state has been completed. A sample of boiling soap is withdrawn from the kettle with a trowel. If the soap is in a grained condition, several drops of clear lye will separate from the curdy mass. If no lye separates from the mass, the kettle is still



in a closed state and requires more salt. If the clear separated lye is dropped on a cold surface of metal or glass and, on cooling, remains clear, it is an indication that graining is complete. If, however, the drop of lye becomes opaque and milky, then additional salt must be added to complete the graining out.

A second method of determining if graining out has been completed is to filter a small sample of clear lye from a mass of curdy soap taken from the kettle. Salt or concentrated brine is stirred into the clear lye. If further curds of soap separate from the clear lye, then graining out is incomplete and more salt is needed for the kettle. If the lye remains clear, graining out is complete.

Oversalting can be corrected by dilution of the kettle with water. However, it is best to avoid oversalting by approaching the end point of the graining operation with care.

A definite change in the physical appearance of the boiling soap can be detected when the end point is approached.

When the kettle is properly grained, it is allowed to settle overnight.

Second Change. The spent lye which has settled out overnight from the first change in Kettle A is discharged to the glycerine department for glycerine recovery. Kettle A is then given its second change. Enough water is added to the curd to just "close" the soap. At the same time, open steam is turned on in the kettle for agitation. When the soap has smoothed out to a pasty, closed condition, fresh caustic soda lye is run into the boiling kettle to complete the saponification of the change. Care must be taken not to add the caustic much faster than it will react with the unsaponified fat. An excessive concentration of free caustic will grain out the soap and retard saponification. Complete saponification can be completed in a relatively short period of boiling in the closed state with but a slight excess of caustic.

When the charge is completely saponified and no longer reacts with a slight excess of caustic in the kettle, soap lye is pumped over from the third change of Kettle D. While this lye is being added, constant agitation with open steam is maintained. The kettle is then grained out with fresh caustic or dry salt and allowed to settle overnight.

Third Change. The soap lye which has settled out overnight from the second change in Kettle A is pumped over to Kettle B which is being given its first change.

Change No. 3 is then given to Kettle A. As in Change No. 2, the curd is closed up with water while being agitated with steam. To make doubly sure of complete saponification, a small addition of fresh caustic is run into the kettle and boiling is continued for a short period while the kettle is in a closed state.

Niger is pumped from storage into Kettle A with constant boiling. The wash is built up to the desired

size and the kettle is grained out with brine or caustic or a combination of the two.

It is to be noted that the relative concentration of NaCl and NaOH on this change will be the same as found in the neat soap after the kettle is fitted and settled. Hence, regulation of the relative NaOH and NaCl content of this change affords a means of adjusting free NaOH or NaCl in the finished neat soap.

When the wash has been built to the proper size and the kettle is grained out, it is allowed to settle overnight.

Fourth Change. The lye from the third change of Kettle A is pumped over to Kettle B for the second change on the latter.

Change No. 4, the "fit," is then given to Kettle A. This is a critical operation and requires considerable experience in its accomplishment since it is difficult to determine beforehand by analytical means the correct amount of water to be added. After the soap lye has been pumped from Kettle A to Kettle B, the curd is boiled on open steam and the proper quantity of water is added gradually to the kettle to obtain the type of fit desired. Webb (3) describes the fitting operation in detail, especially in regard to the types of fit from "coarse" to "fine." Roughly, four or five pounds of water, including steam condensate from the open steam coil, are required for every 100 lb. of normal curd soap to obtain a "medium" fit.

If too little water is added on this change, an extremely coarse fit is obtained which settles out little dirt and produces neat soap which is short in character and unsuitable for further processing.

On the other hand, an excess of water produces a viscous, waxy mass which will settle out a niger only with difficulty. The soap handles poorly, if at all, on further processing.

The trowel test as described by Webb (3) is the best practical guide for the correctness of the operation. This is an empirical test depending on the degree of tackiness possessed by the kettle soap. Tackiness is measured by observing how hot soap dipped up from the boiling kettle slides from a tilted trowel.

A definite correlation can be noted between the way the soap slides off the trowel and the type of fit. In coarsely fitted kettles, the thin layer of soap has a tendency to break up into flakes as it leaves the trowel and the surface of the trowel dries off almost immediately. In finely fitted kettles, the soap is tackier and tends to drop off the trowel without flaking, leaving the surface of the trowel greasy to the touch. In intermediate fits, the soap has less tendency to flake than in coarse fits and the surface of the trowel requires a few seconds longer to dry after the layer of soap has slid off.

With experience, a soapmaker can observe the progress of the fit by the appearance of the boiling mass of soap.

Wigner (4) describes in detail a method of conducting the fit by adding a predetermined amount of water calculated from the proportion of electrolyte, anhydrous soap, and water, present in the curd. This method appears to have good possibilities for quantitative control purposes when properly standardized.

If, by chance, an excessive amount of water has been added on the fit and the soap is too thick, this condition can be corrected by the addition of a small amount of salt or caustic. However, it is best to avoid such a condition since the addition of extra salt or caustic during the fit will produce excessively large nigers.

Day of Cycle	KETTLE A	KETTLE B	KETTLE C
1	CHANGE 1. Charge with fat and lye from Change 2 of Kettle C. Discharge spent lye to Glycerine Dept.	Settle	CHANGE 3. Charge with niger from storage and fresh lye. Discharge lye to Change 2 of Kettle A.
2	CHANGE 2. Charge with lye from Change 3 of Kettle C. Discharge lye to Change 1 of Kettle B.	Discharge neat soap to process and niger to storage.	CHANGE 4. Fitting Operation
3	CHANGE 3. Charge with niger from storage and fresh lye. Discharge lye to Change 2 of Kettle B.	CHANGE 1. Charge with fat and lye from Change 2 of Kettle A. Discharge spent lye to Glycerine Dept.	Settle
4	CHANGE 4. Fitting Operation	CHANGE 2. Charge with lye from Change 3 of Kettle A. Discharge lye to Change 1 of Kettle C.	Discharge neat soap to process and niger to storage.
5	Settle	CHANGE 3. Charge with niger from storage and fresh lye. Discharge lye to Change 2 of Kettle C.	CHANGE 1. Charge with fat and lye from Change 2 of Kettle B. Discharge spent lye to Glycerine Dept.
6	Discharge neat soap to process and niger to storage.	CHANGE 4. Fitting Operation	CHANGE 2. Charge with lye from Change 3 of Kettle B. Discharge lye to Change 1 of Kettle A.

FIG. 4. Three-kettle countercurrent unit schedule.

When the proper degree of fit has been obtained on Kettle A, it is allowed to settle into the two layers, neat soap and niger. The period of settling will vary according to size of kettles, type of fit, and soap base. Large kettles take longer to settle than small ones. Fine fits require a longer settling time than coarse fits. Kettles containing rosin in the soap base require a longer settling period than straight tallow or grease base kettles.

After proper settling, the neat soap layer is pumped to storage or directly to processing. When the neat soap has been entirely withdrawn, the niger is boiled up with open steam, and is pumped to niger storage for reuse in a succeeding kettle.

If the niger is excessively dark, it may be cleaned up by an operation called a "pitch." This is done by diluting the niger with a small amount of water while boiling it with open steam and then allowing it to settle. A dark sludge will settle to the bottom of the kettle. This mass is discarded or, if it contains enough soap, it may be used as soap stock for a low grade soap. The balance of the niger is pumped to niger storage for reuse.

Variations in the Countercurrent System

Three Kettle Unit. It is possible to conduct the countercurrent system in a three-kettle unit if the kettles are small enough in size to require less than two days' settle after the fit. Figure 4 illustrates the six-day cycle schedule for the three-kettle unit. This unit is equal to the four-kettle unit in ease of operation and in efficiency of glycerine recovery and removal of soluble impurities.

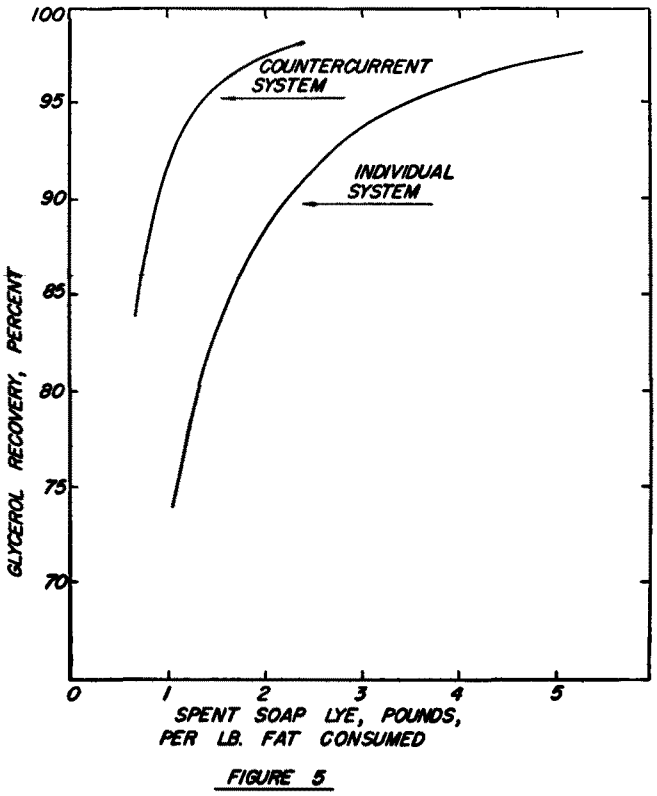


FIGURE 5

Niger Handling. Instead of removing the niger before starting a new boil, the niger can be left in the kettle and the new charge pumped into it. The presence of soap in the niger is an aid to initial saponification in the first change. However, the niger dilutes the concentration of glycerol in the spent lye and increases its bulk. Furthermore, the working capacity of the kettles is decreased by 10% to 15%. Thus, little useful purpose is served by allowing the niger to remain in the kettle.

Glycerol Recovery Comparison

Figure 5 shows a comparison of pounds of spent lye produced per pound of fat charge at varying levels of glycerine recovery for both the individual and the countercurrent wash systems. Each system employs three washes.

Glycerine recovery from the kettles for the individual wash system is derived from the formula:

$$\% \text{ Glycerine Recovery} = \frac{300\,(L+M+N-LM-LN-MN+LMN)}{2+L+M+N-LM-LN-MN+LMN}$$

In this formula, L. M. and N represent the fraction of free glycerol in the kettles removed in each of the three changes.

In like manner, glycerine recovery for the countercurrent system is derived from the formula:

$$\% \text{ Glycerine Recovery} = \frac{200\,L\,(0.9-0.9N+1.4\,MN+0.1M)}{2-2N+2MN-2M+2LM+LMN}$$

The letters L, M, and N represent the fraction of free glycerol removed on the 1st, 2nd, and 3rd changes respectively.

Saponification in the countercurrent system is taken to be 90% complete in the first change and 100% complete in the second change. For simplicity, saponification in the individual wash system is taken to be complete in the first change.

In both systems, the composition of the curd is assumed to be 62% anhydrous soap and dissolved solids and 38% aqueous. The lye layer is taken to consist of 10% dissolved solids and 90% aqueous. On the settle, the distribution of glycerol between the neat soap layer and the niger is taken to be in the ratio two to one.

The ratio of spent soap lye to fat charge for each definite glycerine recovery is calculated using the above compositions for curd and soap lye.

For illustration, the values for L, M, and N used for the countercurrent flowsheet in Figure 3 are .67, .71, and .66 respectively. On substituting these values in the above formula for countercurrent glycerine recovery, the percentage of glycerine recovery from the kettles is found to be 94.7%. The ratio of spent soap lye to fat charge is 61,400 lb. lye to 50,000 lb. fat, or 1.23 lb. to one lb.

It may be pointed out for greater clarity that the fractions, L, M, and N, are determined from the composition of the curd and the soap lye. With reference to the flowsheet in Figure 3, the amount of water plus glycerol on the first change is 27,600 lb. in the curd and 55,200 lb. in the lye. The total water plus glycerol in the kettle is therefore 82,800 lb. The amount in the lye, 55,200 lb., is the fraction .67 of the total amount of water plus glycerol in the kettle. Therefore, the fraction, L, equals .67 in the first change. The fractions, M and N, are derived in the same manner for the other changes.

Other points in the curves are derived in the same manner as in the above illustration. However, it must be noted that in the individual wash system the total amount of spent soap lye produced per kettle is found by adding together the three washes.

An inspection of Figure 5 indicates that a 95% kettle recovery of glycerine is practicable for the full countercurrent system of washes, whereas only 85% to 90% appears to be the practical limit for the individual wash system.

Even at the level of 85% to 90% recovery in the individual wash system, costs of treating and evaporating the spent lye produced are considerably more than for a 95% kettle recovery in the full countercurrent system.

Therefore, it appears that it is advantageous to use, wherever possible, the full countercurrent wash system instead of the individual wash system.

Summary

Soap boiling operations and the various systems of washes used in full boiled soap making are described. A flowsheet and description of a typical four-kettle unit cycle, employing the full countercurrent wash system, is given in detail.

Kettle recovery formulas on a three-wash basis are given for the individual and countercurrent systems. A graph indicating kettle glycerine recovery and pounds of spent lye per pound of fat charged to kettles is also shown for the individual and countercurrent systems.

SUPPLEMENT

It is felt that the derivation of the equation used for estimating glycerol recovery in the full countercurrent system will be of value. This is given below:

Let L = fraction of free glycerol in kettle removed by first wash

M = fraction of free glycerol in kettle removed by second wash

N = fraction of free glycerol in kettle removed by third wash

a = glycerol in niger

s = glycerol bound in unsaponified portion of fat charge in first change

y = glycerol in the lye from a second change

z = glycerol in the lye from a third change

R = fraction recovered of available glycerol in the fat charge

1 = available glycerol in the fat charge

Then,

$$R = L(1-s+y) \quad (1)$$

$$y = M[(1-L)(1-s+y)+s+z] \quad (2)$$

$$z = N[(1-M)[(1-L)(1-s+y)+s+z]+a] \quad (3)$$

Solving (2) and (3) simultaneously, the following value for "y" is obtained,

$$y = \frac{M(1-L+Ls+Na)}{1-N+MN-M+LM} \quad (4)$$

Solving (4) and (1) simultaneously, the following value for "R" is obtained,

$$R = L\left[1-s+\frac{M(1-L+Ls+Na}{1-N+MN-M+LM}\right] \quad (5)$$

Now if it is assumed that the charge of fat is 90% saponified on the first change, then the fraction "s" representing the glycerol bound in the unsaponified fat is 0.1 and (5) becomes,

$$R = L\left[0.9+\frac{M(1-0.9L+Na)}{1-N+MN-M+LM}\right] \quad (6)$$

Now if it is assumed that on the fitting change the free glycerol is divided between the niger soap and the neat soap layers in the ratio of one to two respectively, then since the glycerol in the niger is represented by "a," the glycerol in the neat soap can be represented by "2a."

It then follows that

$$R = 1-2a$$

or

$$a = \frac{1-R}{2} \quad (7)$$

where "R" is the fraction recovered of the available glycerol, "2a" is the glycerol lost in the neat soap, and "1" is the available glycerol in the fat charge. Solving (6) and (7) simultaneously, the following value for "R" is obtained,

$$R = 2L\left[\frac{0.9-0.9N+1.4MN+0.1M}{2-2N+2MN-2M+2LM+LMN}\right] \quad (8)$$

Multiplying both sides of equation by 100,

$$\% \text{ Recovery} = 200L\left[\frac{0.9-0.9N+1.4MN+0.1M}{2-2N+2MN-2M+2LM+LMN}\right] \quad (9)$$

which is the recovery formula for the full countercurrent wash system.

LITERATURE CITED

(1) Ferguson, R. H., Oil and Soap *14*, 115-8.
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Report of the Gossypol Committee

The Gossypol Committee of the American Oil Chemists' Society, since its original appointment in April, 1945, has been engaged in a comprehensive program involving the investigation of the completeness, accuracy, and specificity of the various published methods for the determination of gossypol in cottonseed and cottonseed products.

The first phase of this program, namely, investigation of methods for the determination of gossypol in unprocessed cottonseed, has been under way for some time. The methods listed below are being applied to the determination of gossypol in samples of two lots of pure-bred cottonseed furnished by Dr. J. Winston Neely of the U. S. Cotton Field Station at Stoneville, Mississippi. The methods being investigated cover those pertaining to the extraction of gossypol and subsequent estimation of the content of gossypol in the extracts. The methods are as follows:

Methods for the Extraction of Gossypol from Cottonseed

1. *Exhaustive extraction for 24-72 hours:* Extract in Soxhlet type extractor with diethyl ether (peroxide-free). Schwartze, E. W. and Alsberg, C. L., J. Agr. Res. *25*, 289-295 (1923).
2. *Exhaustive extraction* in Soxhlet type extractor with diethyl ether (peroxide-free) containing 2.3-2.5% ethyl alcohol (by weight) and 1-1.2% water, and having a density of 0.724-0.726 g./cc. at 15.6°C.; water added to mixture in receiving flask (5 cc./350 cc.) [Smith, F. H., Ind. Eng. Chem. Anal. Ed., *5*, 29-33 (1933); F. H. Smith, private communication.]
3. *Exhaustive extraction* in Butt type extractor using same solvent mixture as in (2). Lyman, C. M., Holland, B. R., and Hale, F., Ind. Eng. Chem., Anal. Ed., *15*, 489-491 (1943).
4. *Equilibration:* Equilibrate finely ground seed in chloroform at 37-38°F. according to the method described by Boatner, Caravella, and Kyame, Ind. Eng. Chem., Anal. Ed., *16*, 566-73 (1944).
5. *Equilibration:* Same as (4) using diethyl ether as solvent. Same reference as (4).
6. *Equilibration:* Same as (4) using chloroform at room temperature with agitation according to method of Procter and Gamble, unpublished.