Rapid Settling of Soap in the Course of Laboratory Soap Boiling

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IN the soap industry the rapid conversion of fats into kettle soap on a laboratory scale is extremely desirable. As is commonly known, the fatty raw material may have a relatively light color. However, on conversion into soap extensive darkening may occur. This change in color invariably occurs in the case of fats containing certain phenolic impurities and also if contact with air or other form of oxidation has resulted in formation of α-dicarbonyls or other latent color bodies. On the other hand, a fat may "wash up" well on saponification and may lose as much as 50 per cent, or even more, of its color in the course of lye washing and "fitting" operations. Also other important properties of the soap, such as "life" and hardness, cannot definitely be predicted from the appearance of the stock but can only be satisfactorily determined by conversion of the fat into soap.

In view of this the soap industry has been doing appreciable experimental work on laboratory soap boiling. Very satisfactory procedures have been described by R. E. Divine (1), W. C. Preston (2) and T. H. Wigner (3). These laboratory methods, as well as others published in the literature, utilize gravity settling for the separation of the lyes from the "curds" and for final separation of the "neat soap."

In our experience the settling of soap by gravity on a laboratory scale is not only time consuming, but the results obtained by it are often unreliable. It is obvious that the separation of the nigre, the removal of steam bubbles and the maintenance of a uniform moisture content throughout a small amount of soap are objects difficult to obtain in view of the fact that from 12 hours to several days of heating at about 70 to 85°C. are required to complete the settling. Duplicate samples of soap prepared in this manner almost invariably show appreciable variations in specific gravity and appearance.

In view of this we have proceeded to apply centrifugal separation in the course of laboratory soap boiling. By using a suitable speed of the centrifuge we have found it possible to obtain very close duplication not only between laboratory samples, but also between soaps obtained by processing on the kettle floor and in the laboratory.

Attention is called here to the fact that replacement of gravity settling of soap by centrifugal separation on a commercial scale was first suggested by M. H. Ittner (4).

In laboratory processing we use an unheated centrifuge for the separation of the "curds" from the lyes. For separation of "neat soap" from "nigre" the centrifuge is heated to a temperature of about 90°C. Preferably the entire kettle is submitted to centrifuging. If the capacity of the available centrifuge does not permit this, the kettle contents are rapidly transferred to wide-mouthed hot centrifuge cups and submitted to centrifuging without unnecessary delay.

THE boiling may be advantageously carried out in a conical shaped glass kettle which can be heated in a bath of boiling water and which is provided with open steam as described by Divine (1). For the "kill" the molten fat charge and three-quarters to an equal weight of boiling water containing some sodium chloride are transferred to the heated kettle. 35° Bé. caustic soda, or the lye from a preceding strengthening change, is gradually added to the boiling kettle contents which is well agitated with open steam. The rate of addition of the caustic solution is adjusted in such a manner that at any time, enough electrolyte is present to prevent lumping ("bunching") of the soap, due to formation of middle soap while the excess should not be so great as to throw the soap out of solution. Boiling is continued until any appreciable consumption of alkali has ceased. The soap at this point should have lost its greasy feel. Salt is then added until the soap is salted out. Sufficient electrolyte should now be present to result in complete graining without any possibility of nigre formation. Boiling is continued for about 15 minutes. Separation of grained soap from the lye is then attained by the use of an unheated centrifuge, or by gravity settling. The spent lye should not show turbidity on cooling in the case of satisfactory saponification.

For the lye washes the curds are dissolved in one-half to an equal weight of water which, pending the nature of the fat charge, contains a corresponding amount of salt and free alkali. Heating at about 100°C. and agitation with open steam is continued for one-half hour. Graining is done using excess salt.

In case it is preferred to carry out the lye wash without closing the soap, concentrated salt solution is added to the curds and the kettle contents is boiled for 15 minutes. Separation of the curds from the lye is carried out by centrifuging, using the unheated centrifuge, or by gravity settling.

For the final lye wash which corresponds to the "strengthening change" of regular kettle room practice the curds are again dissolved in water and aqueous caustic alkali is added in small portions. The amount of free caustic present should, at this phase, be nearly sufficient to salt out the soap. If graining should actually take place, a small amount of water is added in order to bring the soap back into solution. Boiling is now continued for about one-half hour. Graining is done using salt or caustic lye. The separation of curds and lye is again attained by centrifuging, using the unheated centrifuge, or by gravity settling.

FOR the finishing operation, known in kettle room practice as "pitching" or "fitting" the curds are thoroughly boiled with open steam. Enough water or 35° Bé. lye is now carefully and very slowly added to give the required "fit." This state is best judged by the manner in which the soap boils and in which

it runs off a hot spatula blade held in a vertical position. The ratio of anhydrous soap to water should now be about 1:1 in the case of a tallow soap. If necessary, final adjustment is made with hot concentrated salt solution.

The finished soap should slide off the spatula in a coherent sheet leaving a thin aqueous film which rapidly evaporates. If the soap runs off too freely, breaking into small strands or flakes and leaving the spatula surface covered with a film of lye, more water is needed. If the soap slides off with difficulty or not at all, more salt is required. For details of the soap boiling process see description by McBain (5) and Webb (6).

When the proper adjustments have been made, separation into "neat soap" and "nigre" or generally into a thin foamy top layer called "fob," next the neat soap, then the nigre and at the bottom a small quantity of lye known as "pitch water" is done by centrifuging for from 5 to 10 minutes at a speed of about 1500 r.p.m. and at a temperature above the solidification point of the soap. The soap is then permitted to cool at room temperature before it is removed from the centrifuge cups.

Figure 1 shows centrifuge used in these experiments with heating unit installed on cover.

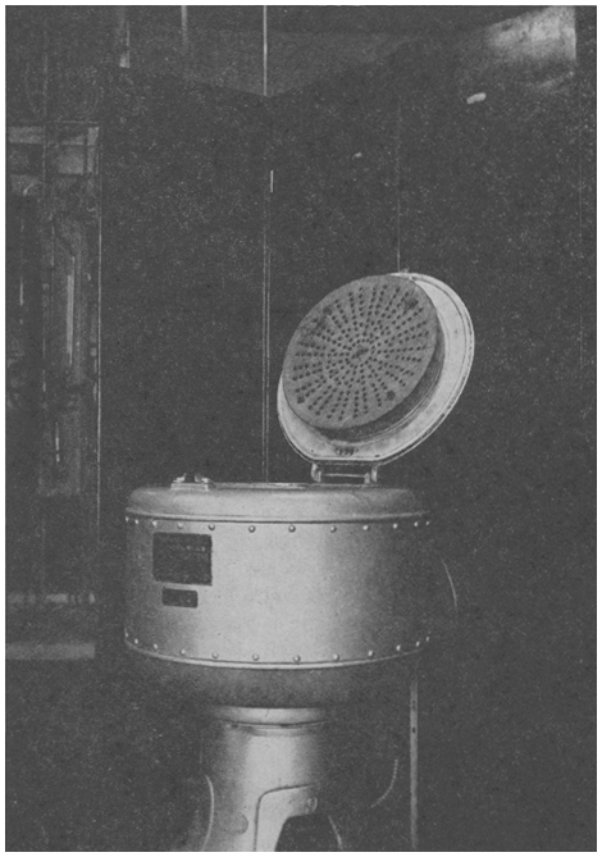
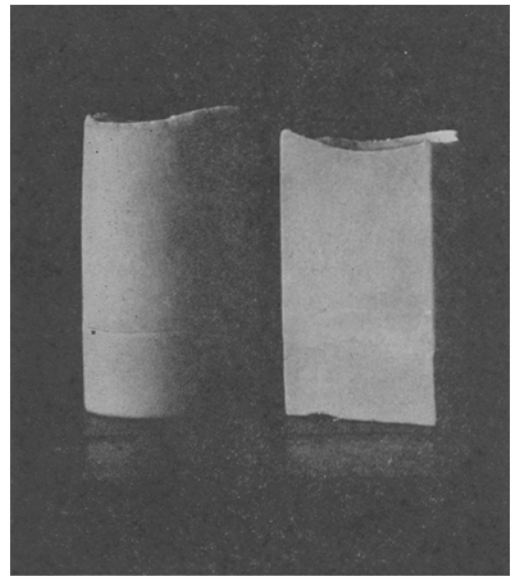


Figure 2 shows the finished soap—nigre separation obtained by centrifuging of a soap made from 90% tallow—10% babassu oil stock. The soap, in this case, had been withdrawn from a 100,000-lbs. saponified and fitted charge just prior to settling for the purpose of checking the extent of duplication that may be obtained by gravity settling and centrifuging.



After the soap in the kettle had been permitted to settle for 96 hours, representative samples of neat soap and nigre were taken. The electrolyte content and the color of the fatty acids were determined on both the centrifuged and gravity settled soaps:

	% Free Caustic as Na_2O	% Na_2CO_3	% NaCl	Color of fatty acids obtained from soap. Lovibond 5¼" cell
Finished soap obtained by gravity settling	0.08	0.11	0.42	4.8R - 48Y
Finished soap obtained by centrifuging (1400 r.p.m.—5 minutes	0.06	0.08	0.40	4.5R - 45Y

	Lovibond 5¼" cell
Color of fatty acids from nigre obtained by gravity settling	43R - 70Y
Color of fatty acids from nigre obtained by centrifuging	45R - 70Y

Summary

A method is described in which gravity settling has been replaced by centrifugal separation in the course of laboratory soap boiling.

The time required for the preparation of a sample of soap in this manner can be reduced to about 8 hours.

Results are well reproducible and by selection of suitable conditions during centrifuging, samples can be made to closely duplicate corresponding soaps made on the kettle floor with regard to appearance and composition.

Acknowledgment

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