United States Patent Office 3,274,119 Patented Sept. 20, 1966

3,274,119

TRANSPARENT SOAP BAR

Seymore Goldwasser, Teaneck, and Frederick V. Ryer, Oradell, N.J., assignors to Lever Brothers Company, New York, N.Y., a corporation of Maine

No Drawing. Filed Mar. 1, 1963, Ser. No. 262,225
7 Claims. (Cl. 252—368)

This invention relates to a transparent soap bar. More particularly, it is concerned with a transparent soap bar with a low moisture content.

It is known to react an ester of glycerol and a fatty acid, such as a vegetable or animal fat or oil, with an alkali metal hydroxide, such as sodium hydroxide, to form a salt which is known in the art as a soap. Although the aforementioned fats and oils, e.g., tallow and coconut oil, differ with respect to the fatty acids therein, they generally have both saturated acid residues, e.g., palmitic acid, and unsaturated acid residues, e.g., oleic acid, with primarily a cis-configuration. Furthermore, a soap may be provided by reacting an alkali solution with a cis-fatty acid, per se.

It is also within the knowledge of the artisan to form a soap bar from a soap stock with the aforementioned reactants therein. The saponification step may take place in a kettle in accordance with the "full-boil" process wherein the saponification is carried out in a series of successive stages with the soap being salted out of the glycerol solution or in accordance with the "Twitchell process" wherein free fatty acids are used. The soap from the kettle is subsequently shredded, partly dried, mixed with perfume, opacifiers, preservatives, etc., milled in roll mills, and plodded into a bar. This end product is known in the art as a milled soap bar.

Although milled soaps have some small degree of translucency, thev are not considered to be transparent soaps. However, transparent soaps are known in the art. Some have enjoyed for many years a limited commercial appeal but they are expensive due to high production costs. The so-called transparent soaps, prepared by cheaper methods of production, are no longer acceptable by present day commercial standards.

A method, which has been employed to produce the more acceptable type of transparent soap, is to prepare a soap base of reduced moisture content and then dissolve this soap in alcohol, removing the saline impurities by decantation. Alcohol is recovered from the soap by distillation. The soap mass then is cooled to solidify and mold. This method is expensive, time consuming and involves the use of high cost solvent.

A cheaper method for manufacturing a transparent soap uses the well known cold process technique. Tallow low in free acid, coconut oil and rosin are saponified with caustic soda in the presence of alcohol or alcohol and glycerin or in the presence of a sugar solution, and the soap mass is recovered and subsequently molded. Another method involves the semi-boiled process and includes crutching the initial oils and fats at approximately 140° F., saponifying with caustic, then adding lye, and stirring until the soap has reached a desired consistency. This is followed by adding sugar dissolved in water, or alcohol and glycerin, or combinations thereof. The mass is again crutched at about 160° F., and the desired perfume and dyestuff are added. The soap mass is molded by framing, then slabbed, cut and pressed. However, neither the cold process nor the semi-boiled process produces soaps of high quality.

In the past, a transparent milled soap bar with a relatively high moisture content, e.g., more than 20% water, has been formed from ordinary fat stocks, such as an admixture of 80% tallow oil and 20% coconut oil (U.S. Pat. No. 2,970,116). It is commercially undesirable to have this high water content since the soap bars are too soft and have a tendency to shrink. As disclosed in U.S. Pat. No. 2,970,116, the moisture content in a soap bar may be decreased to about 17.6% but in order to maintain transparency, the salt content must be similarly reduced. However, a transparent soap bar with an extremely low moisture content, e.g., 14%, could not be provided heretofore, even with a further reduction in salt content.

It has now been discovered in the present invention that a low moisture, transparent soap bar can be provided if the soap stock contains about 5% to about 24% of the acids in the trans-form. The trans-content of a fat is determined from the infrared absorption at about 10.3 microns, and the result is expressed as percent elaidic acid on the basis of the total fatty acids. Furthermore, the instant transparent soap has excellent lathering properties, firmness, a smooth appearance and a waxy feel; and it can be produced without waste or time loss and with minimum cost. For example, there is no solvent used which must be recovered; no additive, such as sugar, rosin, alcohol, and the like remains in the final bar product to detract from its desirable characteristics; and only the usual, available equipment in ordinary soap making is required.

Moreover, the low moisture, transparent soap produced by the present invention has a pleasing firm yet velvety texture. During washing, it does not form a mushy coating on its surface; it does not mar the appearance of the soap dish; and it does not tend to form unsightly cracks. If the instant soap is maintained for considerable lengths of time in contact with water, for example, when an incompletely dry soap dish is used, the contacted portion may become cloudy; however, the soap, upon removal from such contact, will return to its original firmness and transparency. Furthermore, the soap bars of this invention may be used even though worn to wafer thinness.

As is mentioned by F. W. Wells in Soap and Chemical Specialties, vol. XXI, No. 6 and No. 7, June and July 1955, a soap to be transparent must permit boldface type of about 14 point size to be read easily through a ¼ in. section. The terminology "transparent," use to describe the products herein, refers to this standard. Other methods, e.g., "Translucency Voltage," have been employed to evaluate the translucency and transparency of soaps (U.S. Pat. 2,970,116).

However, a method for accurately measuring the transparency of a soap bar has been developed. Three grams of worked soap are weighed into a cylindrical steel mold of one-inch diameter fitted with a tight steel plunger and squeezed in a vise with maximum arm pressure to yield a wafer approximately ⅛-inch thick. The wafer by visual observation may be classified as opaque, translucent, or transparent. The degree of transparency, however, is advantageously determined by inserting the soap wafer between an incandescent light source and a photoelectric cell connected to a microammeter. The intensity of light transmitted therethrough is measured in microampere units (μa). A pressed wafer having a μa value of 25 or lower is considered to be opaque; 25 to 30 is translucent; 30 or higher is transparent. It should be noted that a μa value of 30 corresponds to Wells' stands for transparency. The higher the μa value, the more transparent is the soap.

A low moisture soap bar with the aforementioned transparency is formed by incorporating into a soap stock about 10% to 60% of a fat about 35% of which is in the trans-form. In other words, as low as about 2%, sometimes up to about 3%, and as high as about 21% added trans-acid stock, based on total fat charge, promotes transparency. Since untreated tallow may contain up to 3% trans-acid radicals, the total operable trans-content must be about 5% to about 24%; this range is critical in view of the fact that higher total amounts, e.g., 31%, and lower total amounts, e.g., 3%, do not form a low moisture, transparent product. These trans-acids, e.g., trans-oleic acid, may be provided by any procedure known in the art. A preferred method is to hydrogenate white grease with a sulfurated nickel catalyst as disclosed in U.S. Pat No. 2,468,799 which is incorporated herein as by reference. Other processes for providing trans-oleic acid, which is also known as solid isooleic acid and elaidic acid, are described in U.S. Pat. Nos. 2,165,530; 2,359,404; 2,692,886; and 2,746,979.

The sulfurated nickel catalyst, which is preferred for forming trans-acids, may be prepared by adding flowers of sulfur to refined and bleached soybean oil with the mixture therefrom being heated with constant mechanical agitation. After raising the temperature of the oil and agitating with a stream of carbon dioxide, the sulfurated oil thus prepared may be used in making the catalyst. Refined and bleached soybean oil containing 5.0% of a "spent" (fifth-run) nickel catalyst may be added to the sulfurated oil; the sulfur in the sulfurated oil is advantageously 3.75% based on the amount of nickel in the catalyst. A converter is charged with this mixture of refined and bleached soybean oil, spent catalyst and sulfurated oil, and a hardening operation is carried out until the oil reaches an iodine value of 86 to 90 with subsequent cooling and filtering. The sulfurated catalyst therefrom may contain 3.65% sulfur on the nickel basis. The total amount of the sulfur based on the weight of the catalyst (as distinguished from the nickel in the catalyst) may be .78%. This catalyst suspended in oil is used to hydrogenate at fat or oil to form the trans-acids, e.g., trans-oleic acid.

Although the fat or oil which is treated to isomerize the oleic groups is preferably white grease, tallow oil and palm oil are also suitable. In fact, trans-oleic acid or other trans-acids may be obtained from any fats or oils rich in oleic acid radicals or other unsaturated acid radicals. By appropriate choice of hydrogenating procedures, oils, such as corn, olive, soybean, peanut, cottonseed, sesame, lard, safflower, etc., can be hardened to a desired degree and also isomerized in the same operation to make them suitable for use in the invention. Potassium ion, rosin, and castor oil, well-known transparency promoters, are not excluded from the formulations. However, it is a feature of the invention that these materials are not necessary for transparency since, for example, rosin imparts undesirable color and color characteristics to the soap, and castor oil and caustic potash are relatively expensive.

The auxiliary non-isomerized fats included in the soap stock may be high lauric oils, such as coconut oil, babassu oil, ucuhuba oil, palm kernel oil, or ouri curi oil in amounts from 0% to about 25%. However, an excessive coconut oil content or lauric-myristic content in the soap bar has a detrimental effect on the action of trans-acids in promoting transparency; therefore, as little as possible consistent with good lathering properties is used. The optimum percentage of coconut or equivalent oil is about 20%, but more or less can be used, particularly if the fat formula is balanced to provide desirable bar soap characteristics.

The remainder of the auxiliary fat in the soap stock is preferably tallow oil or palm oil. Other fats, such as hydrogenated fats or oils, may be employed; however, they must be capable of being used in conjunction with

trans-acids and with, or without, high lauric oils to provide a satisfactory commercial soap. For example, about 30% to 40%, based on total acid radicals in the total fat stock or charge, may be saturated C_{16} to C_{18} fatty acid radicals and about 20% to 40% may be cis-oleic acid radicals. A good balance of transparency, lathering, firmness and lasting qualities is obtained with a soap stock of 20% coconut oil, 40% tallow oil and 40% isomerized white grease (about 14% trans-acids).

The soap in its primary form, i.e., before crutching, converting to chips and subjecting to the milling operation, may be made in any suitable manner. For example, (1) a portion of the fat stock can be isomerized and mixed with the non-isomerized portion before saponification, or (2) the entire fat charge can be mixed together and the mixture isomerized to the desired degree and saponified, or (3) fatty acids can be split from either (1) or (2) before saponification and then converted to soap. The amount of free alkali in the soap is not critical in this invention, and an amount normally present in a good quality soap bar can exist in the instant low moisture, transparent soap bar. The preferred amounts are 0.01% to 0.04% or higher, e.g., up to 0.1% or more, calculated as Na_2O; however, it can not be too high or skin irritation may result therefrom. The free alkali content may also be zero, as for example in a superfatted soap, but at least traces of free alkali are preferred to assist in retarding rancidity.

A convenient primary soap, i.e., saponified stock with the trans-acids therein, is neat kettle soap which contains about 28% to about 34%, usually approximately 30% to 32%, moisture. The liquid neat kettle soap is generally subjected to ordinary crutching; free fatty acids, e.g., those from coconut oil, are added and the free alkali, if desired, is reduced to a very low level. This crutching operation may be conducted by any conventional means. During crutching, the mixture is generally in the liquid phase at a temperature above 180° F., preferably above 205° F. The kettle soap, after crutching, has a moisture content of about 28% to 34%, usually about 30% to 32%.

The crutched kettle soap is then dried. Since the water level of the finished soap bar must be in a certain range, the drying is carried out to an extent which permits this range to be obtained readily. Any conventional drying method, e.g., ordinary cabinet drying, may be used; however, the preferred method is flash drying and the especially preferred method is tubular drying as described in U.S. Pat. No. 2,710,057. The soap at the end of the flash drying or tubular drying operation is in the molten state, and it is then chilled and solidified, preferably into flakes. The soap is also in solid flaky form if ordinary cabinet drying is employed.

The aforementioned dried soap mass, e.g., soap flakes, is then subjected to a mixing operation at conditions for providing a working and shearing effect. For example, the temperature of the soap at the beginning of the mixing step should be below about 90° F., preferably from 80° to 85° F. The temperature must not rise above about 110° F., and preferably not above 106° F. during the mixing, assuming that the usual types of stocks, the usual time and the usual equipment are employed. At higher temperatures, the soap will be too soft or fluid to resist the operation of the mixer.

The moisture content and salt content may be adjusted before the end of the mixing step in order to provide a finished product with the required amount of each. Small amounts of various optional ingredients such as perfume, coloring materials, lanolin, resin, sorbitol and preservatives, may be added during the mixing; however, these ingredients are not necessary for producing the desired low moisture, transparent soap bar.

A satisfactory degree of transparency may be obtained during the mixing step particularly in the case of neat kettle soap which has been dried by the tubular or flash

method and chilled rapidly. One method of accomplishing this transparency is the selection of the amount of soap in the mixer and the type of mixer blade to provide a high degree of resistance by the soap to the motion of the blades. This resistance converts the mechanical energy of the mixer blades into heat energy and the desired temperature of the mixture can be obtained without the addition of heat from an external source.

To provide a high degree of transparency, the mixing should be performed for about 10 to 30 minutes with the temperature being raised to between 100° and 110° F., preferably between 103° F. and 106° F. If the temperature is higher than these values, it is detrimental to the obtention of transparency during the mixing step or subsequent steps.

The piston converter described in An Investigation of the Crystalline Phases in the System: Sodium Myristate-Water, Buerger et al., the Journal of the American Oil Chemists' Society, June 1947, pages 193–6, is pertinent with respect to applying mechanical energy to soap to attain transparency. In this system, a container is charged with the soap at the desired temperature, and the soap is worked, e.g., for 10 minutes, by forcing a perforated piston back and forth therethrough. It should be understood, however, that it is not critical for the preparation of a low moisture, transparent soap bar that any particular type of mixer be used, or that any mixing step be employed. The type of mixer is important only when a high degree of transparency is desired during the mixing stage. In commercial production the required transparency is more conveniently obtained during the subsequent milling operation. The time of mixing for merely blending the added optional ingredients, such as perfume, without forming a high degree of transparency may be relatively short, for example, 4 to 15 minutes in a Barbour-Stockwell mixer with thick counter-rotating blades.

The next step in the process to form the low moisture, transparent soap bars of this invention is to mill the mixed soap mass to amplify the working effect. A single pass over two five-roll mills is normally sufficient to accomplish this result. However, a refiner, as described in U.S. Pat. No. 2,005,333 may be used instead of the usual mill rolls and mixer. Soap, not already transparent, becomes transparent during milling, if critical temperatures are maintained and if the soap has the required moisture and salt content. With respect to temperature, the soap mass, as it emerges from the mill must be at a temperature from 100° F. to 110° F., preferably from 103° F. to 106° F.

It is necessary for the success of this invention to adjust the moisture content and salt content at least before the end of the milling or other working in order to form a finished soap having the critical amounts of moisture and salt therein. The salt content in the final soap composition must be in the range of about 0.2% to 0.8%, e.g., 0.5% and 0.6%. Although sodium chloride is generally used, the term "salt" is defined herein to include other known water-soluble, soap-compatible electrolytes, such as sodium carbonate, potassium chloride, sodium sulfate, sodium silicate, and sodium tripolyphosphate. Transparency with this range of salt content can be effected with a moisture content between about 14% and 17.5%, e.g., 15% water. A loss of 2% to 3% water by evaporation may occur during milling and plodding and this loss should be considered when adjusting the moisture content. Accordingly, less water is required for transparency at the same salt level in this invention as compared to the systems previously employed.

The soap which emerges from the milling operation in very thin, flaky layers is then compacted into a bar. As defined herein, compacting is an application of mechanical force on a discontinuous mass of soap in any form, for example, particle, flake, or plastic form, to produce a mass substantially without interfaces. The application of mechanical force may be made by extrusion from a plodder; by compression in a soap or tablet press; or by any other conventional means. However, vacuum plodding is preferred since it prevents air from becoming entrapped therein. The soap bar, which leaves the plodder at a temperature of from 98° to 110° F., preferably from 103° to 106° F., may be cut into individual cakes.

Thus, in accordance with this invention, a transparent soap bar has been formed at the usual milled soap water content, e.g., 14%, and at the usual salt content by incorporating trans-acids, e.g., trans-oleic acid prepared by hydrogen-treating white grease with a nickel-sulfur catalyst, into a soap stock. To provide the transparent product of this invention, a soap mass from the aforementioned stock must be worked during a mixing and/or a milling operation at a temperature between 100 and 110° F., preferably between 103 and 106° F., with the required moisture and salt content. The instant soap bar lasts longer and is more economical to the user. Furthermore, it has other desired characteristics before and during washing, e.g., firmness, smoothness, excellent lathering, velvety texture and the like.

The following examples are submitted to illustrate but not to limit this invention. Unless otherwise indicated, all parts and percentages in the specification are based upon weight.

EXAMPLE I

A first soap stock was provided from 20% coconut oil, 70% tallow and 10% cis-oleic acid. A second soap stock was provided by using trans-oleic acid (elaidic acid of K and K Labs, Long Island City, N.Y.) in lieu of the cis-oleic acid. Sodium chloride (0.5%) was added to each and the following results were noted after piston-processing.

With 10% cis-oleic acid

Percent water:	Appearance
12.6	Opaque.
14.3	Do.
16.0	Do.

With 10% trans-oleic acid

Percent water:	Appearance
13.9	Translucent.
15.8	Do.
16.4	Do.

This example demonstrates that 10% trans-oleic acid in the soap stock produces a transparent product at a low moisture content, e.g., 15.8%. In contrast, an opaque product is formed at a low moisture content if 10% cis-oleic acid is employed therein.

EXAMPLE II

Trans-oleic acid was provided by treating 2500 gms. of refined white grease containing 0.3% active Ni—S catalyst (prepared as per U.S. Pat. No. 2,468,799) with hydrogen under the following conditions:

Minutes	P.s.i.g.	Temperature, ° C.	Refractive Value
0	8	160	44.9
20	5	210	44.6
52	6	205	43.8
60	6	205	43.8
70	8	208	43.7
85	7	209	43.65
145	9	209	43.5
	20		
165	20	210	43.4
195	20	210	43.4
[1] 215	20	210	[2] 43.4
265	20	210	43.2

[1] 0.1% additional catalyst incorporated therein.
[2] Material ER423B taken at this point—36% trans-oleic acid

Soap stocks with different amounts of moisture, coconut oil, tallow and treated grease containing trans-acids (ER423–B) were formed as indicated in Table I. There was 0.5% added sodium chloride in each soap stock.

The transparency of each after subsequent piston-pressing was determined as also indicated in Table I.

TABLE I

Soap Stock	Percentage of Each Component			Transparency With % Water Content							
	Coconut	Treated Grease [1]	Tallow	14%		16%		18%		20%	
				μa.	Visual	μa.	Visual	μa.	Visual	μa.	Visual
A	20	10	70	21	Opaque	32	Transp.[2]	32	Transp	33	Transp.
B	20	20	60	30	Transp	33	Transp	35	do		
C	20	40	40	29	Transl.[3]	35	do	40	do	40	do
D	20	60	20		Opaque		Opaque	38	do	38	do
E	20	80			do		do		Opaque		Opaque.

[1] Containing trans-oleic acid (ER423-B). [2] Transparent. [3] Translucent.

It is evident from this example that even 3.6% added trans-acid (6.6 total trans-acid) in a soap stock will form a transparent product at the low moisture content of 16% and that a transparent product may also be provided with a moisture content of only 14% when the trans-content is 11%. However, an opaque product is formed if excessive amounts of trans-acids, e.g., 28% added (31% total), are included in the soap stock.

EXAMPLE III

White grease was treated with hydrogen, as described in Example II to provide 40% trans-oleic acid therein. Forty percent of the treated white grease was mixed with 40% tallow and 20% coconut oil and converted to a high grade soda soap. The soap was partially dried to a water content of 15.2% and worked in a piston converter. The sodium chloride content was 0.5%.

The μa. of the worked soap was 34 which indicated that a transparent product was formed.

EXAMPLE IV

Sodium soap with a moisture content of 17% and a sodium chloride content of 0.5% was prepared from a mixture of 80% tallow and 20% coconut oil and was worked in the piston converter. The worked product was opaque and had a μa. value of 19.

Examples III and IV show that transparency can be provided in low moisture soap bars by having added trans-acids, e.g., 16% (about 18% total trans-acids), in the soap stock.

Having set forth the general nature and specific embodiments of the present invention, the true scope is now particularly pointed out in the appended claims.

What is claimed is:

1. A process for preparing a transparent soap bar which comprises working a liquid neat soap composition having about 5% to about 24% total trans-oleic acid radicals therein based upon total fat charge to convert mechanical energy into heat energy and to cause a rise in temperature to within a range of about 100° to 110° F. whose moisture content and salt content have been adjusted prior to the end of the working step to provide 14% to 17.5% moisture and about 0.2% to 0.8% water-soluble, soap-compatible alkali metal salt in the finished bar; and compacting the soap composition into bar form.

2. A transparent soap bar prepared by the process of claim 1.

3. A process for preparing a transparent soap bar which comprises working a liquid neat kettle soap composition having about 5% to about 24% total trans-oleic acid radicals based upon total fat charge to convert mechanical energy into heat energy and to cause a rise in temperature to within a range of about 100° to 110° F., at least about 2% to 21% of the total acid radicals being nickel-sulfur-catalyst-isomerized trans-oleic acid radicals whose moisture content and salt content have been adjusted prior to the end of the working step to provide 14% to 17.5% moisture and about 0.2% to 0.8% water-soluble, soap-compatible alkali metal salt in the finished bar; and compacting the soap composition into bar form.

4. A process for preparing a transparent soap bar having a microampere value of at least 30 which comprises working a liquid neat kettle soap composition to convert mechanical energy into heat energy and to cause a rise in temperature to within a range of about 100° to 110° F., said soap composition consisting essentially of about 5% to about 24% total trans-oleic acid radicals based upon total fat charge, 0% to 25% high lauric oils, about 30% to 40% saturated C_{16} to C_{18} fatty acid radicals and about 20% to 40% cis-oleic acid radicals whose moisture content and salt content have been adjusted prior to the end of the working step to provide 14% to 17.5% moisture and about 0.2% to 0.8% water-soluble, soap-compatible alkali metal salt in the finished bar; and compacting the worked soap composition into bar form.

5. A transparent soap bar prepared by the process of claim 4.

6. A process for preparing a transparent soap bar having a microampere value of 30 which comprises working a liquid neat kettle soap composition to convert mechanical energy into heat energy and to cause a rise in temperature to within a range of 100° F. to about 110° F., said soap composition consisting essentially of 20 parts of coconut oil, 60 parts tallow oil and 20 parts of nickel-sulfur-catalyst-isomerized white grease containing 36% trans-oleic acid whose moisture content and salt content have been adjusted prior to the end of the working step to provide 14% moisture and 0.5% sodium chloride in the finished bar; and plodding the worked soap composition into bar form.

7. A process for preparing a transparent soap bar having a microampere value of 34 which comprises working a liquid neat kettle soap composition to convert mechanical energy into heat energy and to cause a rise in temperature to within a range of about 100° to 110° F., said soap composition consisting essentially of 20 parts of coconut oil, 40 parts tallow oil and 40 parts of nickel-sulfur-catalyst-isomerized while grease containing 40% trans-oleic acid whose moisture content and salt content have been adjusted prior to the end of the working step to provide 15.2% moisture and 0.5% sodium chloride in the finished bar; and plodding the worked soap composition into bar form.

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LEON D. ROSDOL, *Primary Examiner.*

JULIUS GREENWALD, *Examiner.*

A. T. MEYERS, *Assistant Examiner.*

UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,274,119 September 20, 1966

Seymore Goldwasser et al.

It is hereby certified that error appears in the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 3, line 41, for "at" read -- a --; line 57, for "color", first occurrence, read -- odor --; column 6, lines 43 and 44, for "Do.", each occurrence, read -- Transparent --.

Signed and sealed this 29th day of August 1967.

(SEAL)
Attest:

ERNEST W. SWIDER
Attesting Officer

EDWARD J. BRENNER
Commissioner of Patents