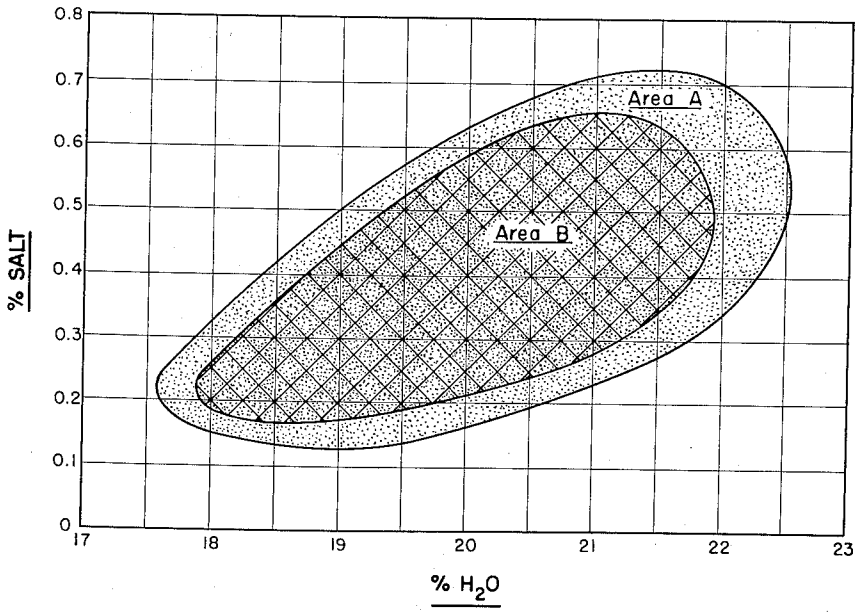
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SOAPMAKING PROCESS

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SOAPMAKING PROCESS

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This invention is concerned with a novel process for the manufacture of transparent soap and with the products obtained by that process which also possess all the desirable properties of the highest quality milled soap.

Although it has been appreciated that soaps ordinarily identified as milled soap have some small degree of translucency, they have no transparency and are not considered by those skilled in the soap art as transparent soaps. Soaps are known in the art, however, as transparent soaps, and some have enjoyed for many years a limited commercial appeal. However, such soaps are expensive due to the method of making them, and those so-called transparent soaps which attempted to use cheaper methods of production are no longer found to be acceptable by present day commercial standards.

One method used in the production of 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 a high cost solvent. A cheaper method for manufacturing a transparent soap used 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 recovered and 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 desired perfume and dyestuff added. The soap mass is molded by framing, then slabbed, cut and pressed. It is well known that neither the cold process nor the semi-boiled process produced soaps of high quality.

Prior art processes for the production of transparent soap are discussed by F. W. Wells in "Soap and Chemical Specialties," volume XXXI, No. 6 and No. 7, June and July, 1955. As is mentioned in these articles, a transparent soap should be sufficiently transparent to permit boldface type of about 14 point size to be read easily through a section a thickness of a quarter inch. It is with reference to this standard that the term "transparent" is used in this application in describing the products of the novel process.

The primary advantage of the method of this invention is that it makes possible for the first time the economical production of a transparent soap having excellent lathering properties, firmness and a smooth appearance and waxy feel. The transparent soap can be produced without waste or time loss and with minimum cost. There is no solvent used which must be recovered. No additive remains in the final bar product such as sugar, rosin, alcohol, and the like which detract from its desirable

characteristics. No unusual equipment not available in ordinary soap making is required.

The 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, and does not mar the appearance of the soap dish. It does not tend to form unsightly cracks, as is the case with many milled soaps. If the soap of the present invention is maintained for considerable lengths of time in contact with water, as happens occasionally when an incompletely dry soap dish is used, it may become cloudy as to that contacted portion, but the soap, upon removal from such contact, will return to its original firmness and transparency. Furthermore, and most surprisingly, bars of soap made by the process of this invention have the very desirable and unique advantage that they may be used even though worn to wafer thinness. Waste is therefore avoided.

Soaps may range from opaqueness through a translucency into true transparency, depending upon the method of manufacture. Various methods have been used to evaluate the translucency, and more specifically the transparency of soaps. A method for accurately measuring this property of a bar of soap is by the use of the following apparatus developed for this purpose. A bar of soap is placed, in a completely darkened room, on top of a cone section surrounding a light source of variable voltage. The cone section has a diameter of ½ inch at the top and 2½ inches at the base, which surrounds the face of the light; the top of the cone section is 9½ inches above the face of the lamp, and the lamp is a microscope lamp with a 120-volt, 15-watt bulb having a blue ground-glass filter. The voltage across the lamp bulb is adjusted until the light from the top of the cone section shines through a bar having a thickness of 2.75 cm. and forms a barely perceptible circular outline. The voltage across the bulb is used as a measure of translucency, which is independent of color and is termed "Translucency Voltage" or "TV." Thus, the lower the TV is, the more translucent the bar. It is possible to measure readily the TV at other bar thicknesses and interpolate to the standard of 2.75 cm. used herein. This method of determining translucency is believed to be superior to a reflectance test described in the art, because it is relatively unaffected by soap color and gloss and avoids the difficulty of cutting a soap bar to a required thickness of only 1/16 of an inch.

For purposes of comparison, an ordinary milled toilet soap of good quality, even in the absence of pigments such as titanium dioxide which make it opaque, has a TV of greater than 110, i.e., it is too opaque to be measured on the apparatus described. This is despite the fact that it has the sheen and glossiness which are commonly referred to as the translucency of milled soap, to distinguish it from the dull nature of frame soaps. The products of the present invention, on the other hand, have a TV of 35 or less, generally 30 or less, when freshly made. A bar of soap with a TV of 30 or less meets the standard required to be called transparent. In general, with the soaps of this invention there is an improvement in transparency upon aging of about six days or more, and in particular it is possible to practice our invention and produce a soap which, when fresh, may have a TV of more than 30, even as high as about 35, which soap, when aged, will acquire the transparent properties of a soap of a TV of about 30 or less. The difference between a bar of soap having a TV of 25 and one having a TV of 35 is quite obvious to the unaided eye.

The most convenient starting material for the present process is soap initially containing from about 28% to about 34%, usually approximately 30% to 32%, mois-

ture, e.g., a neat kettle soap. The precise composition of the initial stock used to prepare the soap is not critical as long as the composition does not differ materially from those ordinarily employed in the manufacture of milled toilet soaps. Thus, for example, the relative proportions of fatty acids from tallow and from coconut oil, and the relative effects of such ingredients on the physical characteristics of milled toilet soap produced therefrom, are well known to soapmakers, and this knowledge is applicable in the formulation of the soap stock used in the present invention. Tests conducted in developing the invention have indicated generally, for example, that the percentage of tallow soap in the soap portion of the final product preferably should be in the range of about 70% to 90%, the remainder being coconut oil soap or equivalent materials. A soap within this range of ratios has good use properties. The ratio of about 75% tallow soap to about 25% coconut oil soap is particularly good.

When neat kettle soap, which is in the liquid state, is used as the starting material, it is first subjected to ordinary crutching, during which free fatty acids, for example those from coconut oil, are added in order to reduce the free alkali to a very low level. This crutching operation is not a critical feature of the invention and may be conducted by any conventional crutching method. The mixture during crutching is, as is conventional, in the liquid phase, at a temperature above 180° F., and preferably above 205° F. The crutched kettle soap, which has a moisture content of from about 28% to 34%, usually approximately 30% to 32%, is next subjected to a drying step. As will be described below, the water level of the finished soap bar must be in a range within certain limits, and the drying is therefore carried out to an extent which permits this range to be readily obtained. The drying step can be carried out by any of the conventional drying methods, for example ordinary cabinet drying. It is, however, preferred to use the method commonly known as flash drying or, most preferably, the method of tubular drying as is described in U.S. Patent No. 2,710,057 of Bassett and Packard. When either flash drying or tubular drying has been used, the soap at the end of the operation will be in the molten state. It is then chilled and solidified, preferably into flakes, before the mixing. There are some advantages, although not critical, in conducting the chilling operation rapidly. When ordinary cabinet drying is used, the soap will be in solid flaky form after the drying.

The next operation is conveniently that of mixing the soap flakes. Mixing provides the opportunity to incorporate such optional ingredients as perfume and coloring matter. It is also a convenient time to adjust the water content, and the salt content. The condition of the soap mass at the time of the mixing operation preferably should be one in which it will permit a working and shearing of the mass to be performed. For example, the soap mass should not be so hot that it is too soft or fluid to resist the operation of the mixer. For this reason, the temperature of the soap at the beginning of the mixing step should be below about 90° F., and preferably should be in the range of from 80° to 85° F. During the mixing of stocks of usual types and with usual mechanical equipment and time, the temperature should not rise above about 110° F., and preferably not above 106° F.

At some point, preferably before the end of the mixing step, the moisture content and the salt content must be adjusted so that in the finished product they will be within the limits which will be discussed below. During the mixing step, small amounts of various optional ingredients are added when desired. These include such substances as perfume, coloring materials, lanolin, resin, sorbitol, and preservatives. The presence or absence of any, some, or all of these optional ingredients is not controlling to the production of a transparent soap bar having the desired characteristics of a high grade milled soap.

Neat kettle soap is a convenient example of the soap stock used. It is, however, not necessary that this be the starting material. What is required is simply a soap mass, however prepared, which is capable of having its moisture and salt contents adjusted to levels which permit the finished soap bar to have moisture and salt levels within the critical range to be discussed below. The drying of neat kettle soap is only one of the possible ways of obtaining such a soap and water mixture. An alternative method is, for example, the reaction between free fatty acids and alkali, to which reaction mixture water is added or removed as required in order to obtain a moisture content within the required range.

Particularly in the case of neat kettle soap which has been dried by the tubular or flash method and subsequently chilled rapidly, a satisfactory degree of transparency is obtainable as early as during the mixing step. To accomplish this, the amount of soap in the mixer and the type of mixer blades employed must be such that the soap offers a high degree of resistance to the motion of the blades; because of this resistance, the mechanical energy of the mixer blades is converted into heat energy, and the desired temperature of the mixture thereby obtained without addition of heat from an external source.

To obtain high degrees of transparency during the mixing, the mixing is conducted for periods of time of about a half hour, the temperature thereby being raised to between 100° to 110° F., preferably to between 103° F. and 106° F. Generally in commercial production it is not convenient to spend this amount of time in mixing, since the desired transparency is more conveniently obtained during the subsequent milling, which need be no more than a nominal amount. The time of mixing generally employed is therefore about 15 minutes, although as little as about 4 minutes is enough to obtain satisfactory blending in of added materials such as perfume or dye. The amount of working required will vary somewhat depending upon the particular soap stock and the particular working device used. The mixing times mentioned above are those for a Barbour-Stockwell mixer with thick counter-rotating blades. It is, however, a matter of routine testing to find the preferred conditions when other types of working are used.

It should be understood, however, that it is not critical to the process of preparing a soap bar of satisfactory transparency that any particular type of mixer be used, or that there be any mixing step at all. Rather, the type of mixer is of consequence only in those cases where it is desired to obtain a high degree of transparency during the mixing stage rather than at a different stage. It should also be understood that in order to obtain a high degree of transparency during the mixing, it is necessary that the mechanical energy of the moving mixer blades be taken up by the soap in the form of heat energy. Thus, a high degree of transparency will not be obtained during the mixing when a mixer with blades which pass through the soap without encountering much resistance is used along with heat supplied externally.

It is detrimental to the obtention of transparency, not only during the mixing but at any subsequent stage, to allow the temperature of the soap mass to be above about 110° F. during the mixing. Preferably the temperature is kept below 106° F.

The working effect of mixing is preferably amplified by subjecting the soap mass to milling. A single pass over two five-roll mills is normally sufficient. In order to obtain a satisfactory degree of transparency, it is critical that as the soap mass emerges from the mill it be at a temperature of from 100° F. to about 110° F., preferably from 103° to 106° F. Soap not already transparent becomes so during milling, provided it has the correct salt and moisture content, and provided suitable

critical temperatures have been maintained. It is to be understood that a refiner of the type described in Patent No. 2,005,333 may be substituted for the usual mill rolls and mixer, assuming always that the same suitable conditions are maintained relative to the soap stock used. It is thus seen that what is required to make transparent soap by the process of the present invention is that a soap mass having the critical salt and water proportions be subjected to working, its temperature raised to within the range of 100°–110° F., preferably 103°–106° F., and subsequently formed into bars. The working may take place during a mixing and/or a milling operation.

One of the functions of the milling operation is that the soap emerges from the mill in the form of very thin, flaky layers suitable for compacting by plodding into a bar form. The remaining steps in the conversion into bar form are not critical features, and are successfully accomplished by any conventional means. There is, however, an advantage to the use of vacuum plodding, since it most conveniently prevents air from becoming entrapped in the soap bar and thereby impeding translucency. It is advantageous that the soap leave the plodder at a temperature of from 98° to 110° F., preferably from 103° to 106° F. After the plodding, the soap is cut into individual cakes by usual means.

As has been mentioned above, it is critical to the process of this invention that, at least before the end of the working, the water and salt contents be adjusted so that the finished soap has a water and salt content within a critical range. During the milling and plodding operation, a certain portion of the water, usually an amount sufficient to reduce its percentage in the mixture by about 2 to 3, may be lost by evaporation, and this loss must be borne in mind when the water content is adjusted.

The interrelationship of the salt and water content of a freshly plodded soap utilizing a conventional soap stock involving approximately 75% fatty acids from tallow and 25% fatty acids from coconut oil is illustrated by way of example in the attached drawing. This drawing in general illustrates by area "B" the relationship of salt and water which permits the production of a transparent soap bar as freshly plodded. Area "A," outside of area "B," illustrates a relationship of salt and water composition which results in a transparent soap having a TV value of 30 after a reasonable period of aging, such as six days. Such soaps lying in area "A" would have a TV value as high as 35 when freshly plodded.

It is to be noted that area "B" shown in the attached drawing is not rectangular but generally oval and represents a range of soap compositions having a water content of from about 17.8% to about 21.9%, and a salt content of from about 0.16% to about 0.65%. Area "A" lying outside of area "B" represents water content limits of about 17.6% and about 22.6% and salt content limits of about 0.12% and about 0.72%. There may be instances in which a soap having a higher salt and higher moisture formulation lying in an area involving a higher moisture and salt content outside of area "A" will acquire a TV value of 30 or less after aging for a period of a month or more. For example, a freshly plodded soap bar of 25% water and a salt content of 0.90% may ultimately acquire a TV value of 30 or less, assuming the other critical steps of the process have been carried out as described above. This particular phenomenon is apparently one of ultimate loss of water by a slow drying out of the soap bar. This is similar to the previously described phenomen that occurs when a bar is soaked in water and becomes cloudy but which after drying returns to the original transparency. This particular area, however, is of no great importance commercially due to the fact that aging may be required for a period as high as several months in order to obtain a transparency having a TV value of 30 or less in the soap bar. Also warped or shrunk bars may result due to loss of moisture.

With a soap having a water content above 25%, however, milling and plodding are not effectively performed because the soap is too fluid unless its salt content is very high, that is, above about 0.95%. Bars of sodium soap having a salt content above about 0.95%, however, never, even upon prolonged aging, develop transparency even when otherwise made in accordance with the process of this invention.

The areas in which the salt and water content should lie to obtain a transparent soap are shifted somewhat upward and to the left, i.e., toward a range of higher salt content and lower moisture content, when a portion of the soap, say from 5% to 25%, is potassium soap instead of sodium soap.

When in the specification and the claims the term "salt" is employed, particular reference is made to sodium chloride, but it is also intended to include other water-soluble, soap-compatible electrolytes such as sodium carbonate, potassium chloride, sodium sulfate, sodium silicate and sodium tripolyphosphate. There is some evidence to indicate that when the salt is sodium tripolyphosphate, transparent soap is obtained when the areas "A" and "B" in the attached figure are shifted somewhat to the left, i.e., toward somewhat lower moisture contents.

Throughout the entire specification and in the claims, all percentages are percentages by weight.

The following examples are given solely for the purpose of illustration and are not to be deemed limitations of this invention, many variations of which are possible without departing from the spirit or scope thereof.

Example I

Kettle soap, the soap portion of which contained approximately 75% tallow sodium soap and 25% coconut oil sodium soap, the mixture containing about 0.25% sodium chloride and an alkalinity of 0.15% expressed as sodium oxide, was charged into a crutcher, and the free alkalinity reduced to between 0.025% and 0.04% by the addition of coconut fatty acid. During the crutching, which was at 205° F., the moisture content of the soap was adjusted to 30–32%.

By the tubular drying method of U.S. Patent No. 2,710,057, the moisture of the soap was reduced to between 19–20%. The soap was then chilled rapidly on a chill roll to 80–85° F. The dried chips were weighed into a 900–1000 pound mixer fitted with thick counter-rotating blades. 2% water and sufficient sodium chloride to adjust the finished bar to 0.4% salt content were added. The batch was then mixed for 15 minutes. During this period, the temperature of the soap rose from 80–85° F. to 100–105° F. without the addition of external heat. During this same 15 minute period, the soap was converted to a partially transparent state. As the batch was being mixed, the moisture of the soap was determined, and adjusted to 22% water by the addition of water.

The mixed batch was then milled by a single pass over two five-roll mills. The clearances between the mills were 0.040, 0.025, 0.015 and 0.010 inch in the first mill and 0.025, 0.015, 0.010 and 0.005 inch in the second mill. The temperature was controlled so that the soap left the second mill at a temperature between 100 and 106° F. At the end of the milling, the soap was highly transparent. The soap was then plodded into a continuous bar using a vacuum plodder, the extrusion temperature being 103°–106° F. The moisture content of the finished bar was 19.[illegible]%, and it had a TV of 28. As freshly prepared, bars of soap made by this procedure consistently had a TV no greater than 30, generally between 26 and 28, and after about 6 days aging, a TV of between about 20 and 25, generally nearer 20.

Example II

The procedure of Example I was followed, except that during the crutching, 0.1% of the sodium salt of ethylene diamine-tetracetic acid was added as a preservative, and during the mixing the following ingredients were added: 0.7% fern cologne-type perfume, 1.0% lanolin, and 0.0016% Monastral Fast Green pigment. The TV of the finished product was the same as the product in Example I.

Example III

The procedure of Example II was followed, except that besides the ingredients added in Example II, 1.0% Staybelite resin and 0.7% sorbitol were also added during the mixing. The TV of the soap made by this method was slightly improved over that of Example I and Example II when the products were compared in the freshly made state, but when compared after about a week of aging, they were substantially the same.

Example IV

The procedure of Example I was repeated, except that the mixing was continued for 30 minutes, instead of for only 15 minutes as in Example I. By the end of the 30 minutes of mixing, the temperature of the mixture was 106° F., and the soap was transparent. It was then plodded in a vacuum plodder without milling, and the freshly made bars had a TV of 30 or less.

Example V

Kettle soap, the soap portion of which contained approximately 75% tallow sodium soap and 25% coconut oil sodium soap, the mixture containing about 0.25% sodium chloride and an alkalinity of 0.15% expressed as sodium oxide, was charged into a crutcher, and the free alkalinity reduced to between 0.025% and 0.04% by the addition of coconut fatty acid. During the crutching, which was at 205° F., the moisture content of the soap was adjusted to 30–32%.

By the tubular drying method of U.S. Patent No. 2,710,057, the moisture of the soap was reduced to between 19–20%. The soap was then chilled rapidly on a chill roll to 80–85° F. The dried chips were weighed into a 900–1000 lb. mixer fitted with thick counter-rotating blades. 2% water and sufficient sodium chloride to adjust the finished bar to 0.4% salt content were added. The batch was then mixed for 3 minutes, at the end of which time the temperature was still in the 80–85° F. range.

The mixed batch was then milled by a single pass over the two five-roll mills previously described. During the milling, the mechanical energy of the rollers was converted into heat energy, and the temperature of the mills was controlled so that the soap rose from the original 80–85° F. to 103–106° F. At the end of the milling, the soap was highly transparent. It was formed into bars by plodding.

Example VI

The procedures of Examples I through V, inclusive, were all carried out repeatedly, the only changes being in the water content and the salt content of the finished soap bar. When the water and salt contents were within the area "A" of the attached drawing, the freshly prepared bars had a TV of 35 or less, and when the water and salt contents were within the area "B" of the attached drawing, the freshly prepared bars had a TV of 30 or less. Similar results were obtained using sodium carbonate, potassium chloride, sodium sulfate, and sodium silicate as the salt.

Example VII

The procedure of Example I above was repeated, but the salt content of the finished bar was 0.9% and the moisture content was 24%. Bars of soap freshly made by this procedure had a TV of about 47, but after aging for about 6 months, the TV was less than 30.

Example VIII

To illustrate the criticality of the limits of water and salt content, the procedures of Examples I through V, inclusive, were repeated, but the water content of the finished bars was 20%, and the sodium chloride content was 1.0%. The bars all had TV values above 110, i.e., they were too high to measure by the method described. Although they had luster, they were not truly translucent, but were actually opaque.

Example IX

A soap was prepared and crutched as in Example I. It was then cabinet dried to a moisture content of 20.5%. The dried soap was then mixed and milled as in Example I. The product was highly transparent.

Example X

The procedure of Example I was repeated, except that the soap was milled at 94° F. and extruded from the plodder at 93° F. As freshly made, the soap bar had a moisture content of 19.7%, but its TV was 44.

Example XI

The procedure of Example I was repeated, except that the soap was milled at 120° F. The finished bar was opaque.

It is to be understood that various modifications of the method and product described herein will readily occur to those skilled in the art. All such modifications are intended to be included within the scope of the invention as defined in the accompanying claims.

This application is a continuation-in-part of copending application Serial No. 456,148, filed September 15, 1954, by William A. Kelly and Harry D. Hamilton and now abandoned.

What is claimed is:

1. A process for making a transparent soap bar having a translucency voltage of no greater than about 35, said process comprising working below about 110° F. a toilet soap mass having a very low level of free alkali wherein the mechanical energy is converted into heat energy by working to an extent great enough to cause the mixture to rise in temperature to within a range of from about 100° F. to about 110° F., the moisture and water-soluble, soap-compatible, alkali metal salt content having been adjusted prior to the end of the working step to lie within the combined areas "A" and "B" of the drawing hereof in the finished bar, and plodding the soap mass into bar form.

2. A process for making a transparent soap through which mass one-fourth inch thick, a 14-point boldface type is readable, said process comprising working at a temperature above 90° F. and below 110° F., a toilet soap mass having a very low level of free alkali whereby heat is generated throughout said mass by such working, reducing said mass at a temperature between 100° and 110° F. to a form suitable for plodding, plodding said soap mass into a bar form, the moisture and water-soluble, soap-compatible, alkali metal salt content of said mass having been adjusted prior to plodding to a range within combined areas "A" and "B" of the drawing hereof.

3. A process for making a transparent soap having a translucency voltage of less than 35, said value being based upon the voltage required to transmit sufficient light from a 15-watt, 120-volt microscope lamp through a blue ground-glass filter at a distance of 9½ inches, to penetrate a 2.75 cm. thickness of said soap and form a circular outline therein, said process comprising reducing the moisture content of a molten neat kettle soap composition having a very low level of free alkali to substantially less than 22.6% moisture and a water-soluble, soap-compatible, alkali metal salt content to substantially less than 0.72%, working said soap mass whereby the temperature rises to not more than 110° F., reducing the

mass to a ploddable form, and plodding into bars while maintaining the temperature substantially uniform, whereby a waxy textured, transparent, isotropic, microcrystalline soap is obtained.

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

5. A process for making a transparent soap through which mass one-fourth inch thick, a 14-point boldface type is readable, said process comprising working at a temperature above 90° F. and below 110° F., a toilet soap mass having a very low level of free alkali whereby heat is generated throughout said mass by such working, reducing said mass at a temperature between 100° and 110° F. to a form suitable for plodding, plodding said soap mass into a bar form, the moisture and water-soluble, soap-compatible, alkali metal salt content of said mass having been adjusted prior to plodding to a range within area "B" of the drawing hereof.

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

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