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R. H. FERGUSON ET AL
DETERGENT PRODUCT HAVING MILLED SOAP PROPERTIES
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Fig. 1.

Fig. 2.

Fig. 3.

Fig. 4.

Fig. 5.

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This invention relates to soap products which have high water and salt contents and yet have milled soap characteristics, and to a method of making them.

Milled soaps possess easily recognized characteristics which distinguish them from other commercial types of soap and which make them desirable for many purposes. Among these characteristics are a uniform texture, firm consistency, a smooth, waxy feel, surface glossiness, grain or crystal orientation substantially in one direction, transluency (when opaque whitening agent is omitted), freedom from warping or distorting as the bars age and dry out, tendency to swell when soaked in water and yet to return to substantially the original shape and appearance after drying, rapid rate of solution in water, especially when rubbed, and consequent profuse lathering power. Other characteristics, to be discussed hereinafter, are the presence of soap in the beta crystal phase, and the lowering of the "thaw point" of the liquid in the bar.

Milled soaps are conventionally made by "millling" a low moisture, low electrolyte soap (e.g., 10-15% water and 6.4-6.8% salt), that is by passing the largely solidified but still plastic soap between a series of rotating rolls, successive members of the series rotating at higher speeds and at closer clearances, the soap being thus subjected to mechanical working, shearing and compacting. The emerging sheets or ribbons of soap are then further mechanically worked, with shearing and compacting, by being forced through one or more orifices of restricted size by the pressure of an Archimedean screw revolving within a closed barrel. The action of the screw in compressing the soap and forcing it through the orifice is known as plodding and extruding. The mechanical working results in a rise in temperature, which may be regulated by adding or abstracting heat as desired. The combined effect of temperature and of extrusion under pressure is to weld the soap together and cause it to emerge from the orifice as a homogeneous, coherent, slightly plastic bar, which is then cut into suitable lengths and stamped for use.

Hitherto, milled soaps have been made only from soaps which are low in water and in electrolyte content. The neat soap of the kettle, i.e. the so-called "kettle soap" obtained according to conventional soap-boiling practice by settling over nigre, contains about 0.4% or more of sodium chloride and up to about 0.1% of sodium hydroxide or Na₂CO₃, but for milling purposes it is excessively high in water, normally running about 30-52% H₂O. Milling of such soap has been unsatisfactory because of the stickiness of the flakes, the softness of the extruded bars and their lack of the desirable milled soap characteristics. A drying process is therefore commonly practiced before milling, whereby the water content is reduced to about 10% to 15%, but this of course adds to the expense of the soap manufacture.

It is an object of this invention to provide a milled type of soap of high moisture content and a process for making same. It is a further object to prepare such milled type soap from soap originally of high moisture content without incurring an expensive drying step. It is a further object to prepare soap flakes and bars of high moisture and electrolyte content yet possessing milled soap characteristics. It is a further object to provide built soaps possessing the properties of milled soaps. Other objects will become apparent in the description which follows.

We have found that the above and other objects can be attained by mechanically working and compacting (i.e., by subjecting to shearing forces under pressure) a plastic solid mixture of soap, water and electrolyte, the relative proportions of the constituents and the temperature being controlled in such manner, as hereinafter described, as to yield a product of the desired properties. Generally speaking, the soap of our invention may be characterized as a novel milled soap composition which differs from previously known milled soap in a materially increased content of both water and salt or other suitable electrolyte. Our invention opens up a broad new range of compositions for the production of soap products having very desirable characteristics.

The attached drawings are Figs. 1 to 5, in which graphs are given showing the proportions of water and of electrolyte present in the detergents of our invention, the electrolyte in Fig. 1 being sodium chloride, that in Fig. 2 being tetradsodium pyrophosphate, that in Fig. 3 being pentasodium tripolyphosphate, that in Fig. 4 being trisodium orthophosphate, and that in Fig. 5 being sodium silicate.

We shall first describe our invention as if it were restricted to a ternary mixture of soap, water and electrolyte in which the soap is the sodium soap of a mixture of 80% tallow and 20% coconut oil, and in which the electrolyte
is sodium chloride. Later we shall outline our invention more broadly.
The soap for our process can be made by the familiar boiled process in the kettle, with purification by settling and separating into immiscible phases, or by the semi-boiled or cold processes, well known to those skilled in the art, or by continuous saponification of fatty acids under pressure as described in U. S. 2,159,397, or any other method desired. The soap can be adjusted to the desired water content by any suitable means, or it can if desired be used without adjustment of water content if in the manufacture of the soap a suitable water content is attained. If any water is to be removed, obvious means may be employed, such as partial drying of flakes in a drying chamber or spraying the molten soap at high temperature and pressure into an evaporation chamber at lower pressure whereby water is vaporized, conditions being so controlled as to give the desired water content. Alternatively, partially dried soap can be mixed in suitable proportion in a crutcher with molten undried soap until uniform and homogeneous and of the desired water content. If, on the other hand, the water content of the original soap is too low, this can be remedied by crutching a water solution of electrolyte, such for example as sodium silicate, into the molten soap before cooling and solidifying same. Electrolyte can be added to the soap as required, either in dry form or in solution; we preferably, but not necessarily, mix it with the molten soap in a crutcher.

TREATMENT IN THE PLASTIC SOLID STATE

As herebefore stated, our process involves mechanical treatment of a soap-salt-water system which is in a plastic solid condition, deformable under pressure but substantially incapable of flow under the action of gravity alone or of welding together on contact after severance unless considerable pressure is applied. The desired state of plasticity can be attained by any suitable means, such for example as by cooling to a eutectic or by cooling in a thin film over a cold roll or by cooling with agitation as for example in a continuous cooling device (e.g. a "Votator") such as that described in U. S. 2,295,596, in which case the soap is conveniently extruded as ribbons, sheets, threads or cylinders of small diameter, depending upon the size and shape of the orifice or orifices. Furthermore, it is not in all cases necessary that the soap-salt-water system be homogeneous at the beginning of the mechanical treatment, since by such treatment a heterogeneous system can in many cases be made homogeneous. The following discussion will aid in understanding the plastic solid condition contemplated in our invention.

It has been proposed in the past that bars of soap as normally encountered in the household are completely solid, the water content thereof entering into the crystal structure in the manner of water of hydration. More recent research leads us to believe instead that at ordinary temperatures of use, bars of soap, unless extremely dry, are commonly composed of solid soap crystals enmeshing a small amount of a dilute liquid solution of soap in water, known as milk, and that only when chilled to some temperature below 0° C. does this liquid freeze to ice and the bar becomes wholly solid. At temperatures which are sufficiently low, even though they may be above the melting point of the ice, such bars are brittle and friable, while at higher temperatures they are more plastic due to a partial conversion of soap crystals into liquid.

On further warming to well below the room temperature, the proportion of crystals becomes progressively less, and the bar gives place to another fluid phase which (depending on the soap composition and the temperature) may be either next soap of moderate viscosity or middle soap of high viscosity. As the temperature of this two-phase, solid-liquid system rises still further, more and more solid crystals are converted into fluid and eventually the solid phase disappears entirely.

We are not concerned with a wholly fluid system, nor do we practice our invention at temperatures at which the soap is of a pasty consistency such as characterizes a mixture composed substantially of soap crystals and next soap, as in Mills' U. S. Patent 2,295,584, or at a temperature such that the soap is sufficiently soft to be extruded at low pressure, as for example the bars about 5 to 25 lbs. per square inch as in Utten's U. S. Patent 2,377,432. In such systems the fluidity is too great to permit applying the requisite shearing forces, and the resulting soaps, which are free from the shear stresses and internal cleavage planes which are characteristic of the results of milling, plodding and extruding soaps in the plastic solid condition, are also lacking in important milled soap characteristics. In order to produce these properties in our high moisture, high salt compositions, they must be subjected to shearing forces resulting from differential movement in the plastic solid state under high pressure, such as the pressures exerted by milling rolls, or plodding and extrusion pressures of the order of about 100 lbs. or more per square inch. We prefer to operate at a temperature at which our soap-water-electrolyte system is composed of a large proportion of soap crystals and a small proportion of dilute nigre, since in such systems milled soap properties can be attained most easily and to the highest degree. However, in some systems containing a large proportion of crystals and a low proportion of middle soap, the viscosity or plasticity is such as to permit setting up the shearing forces and pressures of our invention and the production of milled soap properties thereby. In general, therefore, we practice our process at temperatures at which next soap cannot exist in stable equilibrium, i.e. at temperatures below the solidification temperature of the system but above the temperature at which it becomes brittle, and such that the system is in a plastic solid condition composed essentially of a mixture of a large proportion of solid soap crystals with a small proportion of a fluid soap phase which is stable at such temperatures and which may be either nigre or middle soap.

The minimum temperature at which heat soap can stably exist can be determined for a given soap composition in a plurality of ways, one way being the well known dilatometric method, since simultaneous appearance or disappearance of next soap is accompanied by a change in slope in the volume-temperature curve of the system. Satisfactory mechanical working and compaction of the soap for the purpose of our invention can take place only when it is substantially in the plastic solid condition as described above. Our process comprises subjecting it, in this condition, to a mechanical kneading, shearing and compacting treatment, such for example as it receives when passing between conventional milling rolls.
but any other means of giving it such treatment can be employed. The action of the milling rolls can be supplemented by the compacting and working action of plodding and extruding. In fact, oft-repeated high pressure extrusions through small orifices accomplish a mechanical working, shearing and compacting similar in nature and in effect to passage through a series of millings rolls, due to the linear flow of the soap under pressure, presumably with adjacent layers flowing at different relative speeds. It will be understood, therefore, that our process is capable of producing soap of milled characteristics in flake form by passage over and between milling rolls or by repeated extrusion through a narrow slit, or in bar form by milling, plodding and extruding, or in other forms such as threads or spaghetti-like noodles depending upon the size and shape of the extrusion orifice.

CRYSTAL PHASES IN SOAP

For an adequate understanding of the results produced by the mechanical working and compacting of our invention, a brief discussion of phase relationships in soap is required. Solid commercial soap is known to exist in at least three distinct crystalline phases, designated beta, delta, and omega, which are convertible into one another. These phases appear either in the finished product or at some stage in the manufacturing process, sometimes existing alone, sometimes in mixtures with one another. Generalizing broadly, it may be said that beta phase is favored by mechanical working of relatively low moisture soaps of usual formulation at suitable temperature, that omega is favored by quietly cooling such soaps from a molten condition in the absence of agitation, and that delta is favored by high molecular weight, and by high water content and low temperatures, as for example when soap nigris solidify.

Differences in crystalline structure are reflected in differences in physical properties for the phases. Ferguson, Rosevear and Stillman (Ind. Eng. Chem., 35, 1005 (1943)) give data illustrating this for three samples of the same soap, each sample having been processed differently in order to yield a different crystal phase. Their data show soap which is in the beta phase as a result of milling and plodding, to be much firmer and more readily soluble, and to lather more easily, than either of the above mentioned phases, and to be distinguished by its greater tendency to swell when soaked in water. Their milling and plodding produce the beta phase under conditions tending to orient and press the crystals together. If the conditions of producing the beta phase are such that the crystals are not packed, joined coherently, or oriented in parallel fashion (as for example by chilling and mechanically working the semifluid soap while in a state of paste consistency) the ease of lather is retained, but optical discontinuities occur, translucency and the characteristic waxy milled charactaristics are lost, and the bar tends to disintegrate in water and to be in general much softer.

The various crystalline phases of soap can be distinguished from one another and identified by certain characteristic X-ray diffraction rings which for a given phase remain practically unchanged by variations in fat formula, water content or electrolyte content, although such variations may be determinative of the phase which forms. A simple system of identification is based upon the characteristic short spacings shown in

<table>
<thead>
<tr>
<th>Phase Diameter (cm)</th>
<th>Lattice Spacing d (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beta</td>
<td>0.35</td>
</tr>
<tr>
<td>Delta</td>
<td>0.40 and 0.65</td>
</tr>
<tr>
<td>Omega</td>
<td>0.35</td>
</tr>
</tbody>
</table>

Table I.

IDENTIFYING X-RAY DIFFRACTION RINGS

Table II.

<table>
<thead>
<tr>
<th>No. of Times Extruded</th>
<th>Phases Composition (by X-ray)</th>
<th>Rate of Wear (Rotary Brush) (Arbitrary Units)</th>
<th>Firmness (Mullen) (lbs.in.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Percent beta</td>
<td>Percent omega</td>
<td></td>
<td></td>
</tr>
<tr>
<td>2</td>
<td>15</td>
<td>65</td>
<td>2.2</td>
</tr>
<tr>
<td>5</td>
<td>30</td>
<td>70</td>
<td>3.5</td>
</tr>
<tr>
<td>10</td>
<td>50</td>
<td>50</td>
<td>4.6</td>
</tr>
<tr>
<td>15</td>
<td>70</td>
<td>30</td>
<td>5.2</td>
</tr>
<tr>
<td>20</td>
<td>90</td>
<td>100</td>
<td>6.1</td>
</tr>
<tr>
<td>25</td>
<td>115</td>
<td>150</td>
<td>6.8</td>
</tr>
</tbody>
</table>
Although the mechanical working and pressure treatment of our process converts the high-moisture, high-electrolyte soaps of our invention predominantly into the beta phase, and although existence of such soaps predominantly in this phase is essential to possession by them of milled soap properties in characteristics degree, yet it is not to be understood that mechanical working of any soap necessarily converts it into the beta phase; for example a soap containing only 6%-7% water and existing predominantly in the beta phase has under suitable extrusion conditions been converted into the omega phase, with resulting decrease in firmness, rate of wear, lathering power, etc., and at high moisture, in the absence of added electrolyte, a framed bar in the beta phase can be converted by extrusion or plodding into a soft delta bar. Furthermore, mechanical working of omega does not per se markedly increase lather; it is when such working effects conversion to beta that improvement results, and this is dependent both upon the composition of the soap and upon the temperature, duration and degree of working and compacting.

**BASIS FOR CLASSIFYING OUR SOAP AS A MILLED SOAP**

As pointed out above, the presence of a proportion of soap in the beta phase is indispensable to the possession of milled soap properties. Although in some cases commercial milled soaps of the past have contained less than 50% of the beta phase, presumably due to incomplete milling, yet in order to have milled properties to the degree contemplated herein, and such that the product can be unequivocally recognized as a milled soap, about 50% or more of the soap in product must be in the beta phase. However, the typical firmness, translucency, glossiness, waxiness, reversibility in water, etc. of milled soap may be lacking even in soap which is predominantly beta phase. For example, soap made by the Mills process according to U. S. Patent 2,295,594 is largely beta, but it is lacking in those milled soap characteristics, despite its high lathering power and its high beta content. In that process, molten soap of suitable composition is rapidly chilled and agitated while in a state of pasty cohesiveness. The product of that process is softer and more opaque than a milled soap.

With respect to milled soap characteristics, it is in some cases hard to compare our products quantitatively with soaps of the same composition obtained by other processes because it is in some cases impossible to make coherent, homogeneous bars of high moisture and high salt content by any other process. In certain other high moisture, high salt compositions, coherent, homogeneous bars can be formed by other processes (as for example, by framing or by the method of Ittner's U. S. Patent 2,377,424), but these bars are lacking in the milled soap characteristics of gloss, translucency, firmness and reversible swelling in water. In the case of the high moisture, high salt compositions which are susceptible to preparation in bar form by methods other than ours, direct comparison of bars so formed with our bars with respect to gloss, translucency, firmness, and reversible swelling in water will show the effectiveness of our shearing and pressure treatment in producing milled soap characteristics.

In determining translucency, the method described in Mills' Patent 2,295,594 is satisfactory, opacity rather than translucency being measured for reasons of convenience. The procedure is to cut from the soap a slice \( \frac{1}{4} \) inch thick, and to measure with a reflectometer the amount of light reflected under standard lighting conditions (a) when the slice is placed on a black background, and (b) when the slice is placed on a white background. The first reading multiplied by 100 and divided by the second reading we call the opacity value of the soap, this being the reciprocal of the translucency. In the example given in the Mills' patent, the product of that process had an opacity value of 89 as compared with 70 for the same soap when it was milled, despite the fact that X-ray diffraction patterns showed both soaps to be of the same beta content. Opacities as low as 27 have been obtained in practicing our invention, the values normally running between this general range and the range of about 70 given by Mills for a milled low moisture, low electrolyte soap.

The firmness of bars can readily be measured with a Mullen tester. Table III shows illustrative opacity and firmness data on two samples of the same soap (80% tallow, 20% coconut oil), one sample being framed (i.e. allowed to solidify by slow cooling from the molten state without agitation) the other being extruded until it possessed marked milled soap properties.

<table>
<thead>
<tr>
<th>Table III</th>
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<tbody>
<tr>
<td>Percent NaCl</td>
</tr>
<tr>
<td>Percent BaO</td>
</tr>
<tr>
<td>Firmness (Millic)</td>
</tr>
<tr>
<td>Opacity, Percent.</td>
</tr>
</tbody>
</table>

To determine rate of solution, a bar of the soap in question can be rubbed in uniform manner with a wet sponge and the amount of soap rubbed off per stroke can be determined. In like manner one can determine case of lathering by counting the number of rubs with a wet sponge necessary to raise the soap concentration in a fixed volume of water of standardized hardness to the point at which stable suds can be observed. Studies of the freezing and melting behavior of the liquid present in soaps show useful correlation with milled soap properties. When soap in the opaque, non-milled condition, whether originally in omega, beta or delta phase, is subjected to shearing forces and high pressure so that milled soap properties are produced, there is a change in state of at least a portion of the liquid which is present in the system. This change is interpreted as indicating that the liquid, which at normal temperatures is present as an isotropic dilute soap solution, is by the mechanical working and compacting brought into a more intimate association with the solid soap crystals which are present, becoming more closely and tightly "bound" thereto by adsorption forces. Its vapor pressure is lowered and its freezing behavior is altered. The changes which take place are most easily detected and followed by dilatometric measurements, which show the changes in volume which occur on gradually warming such systems. The soap is first frozen by thrusting into a bath of alcohol and Dry Ice at a temperature of about -70° C. The phase of the soap crystals not being affected by this treatment. X-ray diffraction patterns on such frozen systems show not only the rings characteristic of the soap, identifying its phase, but also rings characteristic of ice, showing that water...
In the system is frozen. On gradually warming the frozen system, there is first an almost linear expansion caused by resumption of linear expansion with rising temperature, due to expansion of the co-existing soap solution and soap crystals. The sharp inflection of the curve, amounting to a change in sign of the volume change, marks the disappearance of the last trace of ice, and the temperature at which this occurs we shall call the “thaw point.”

In solid soaps containing more than about 40% water, the thaw point approaches the freezing point of water, but with decreasing water content, the thaw point decreases and the deviation from 0° C. becomes easily measurable. More conversion from one phase to another, as for example from omega to opal beta phase, is not accompanied by a change in thaw point, but the shearing-pressure treatment of our process which results in milled soap properties of our products is accompanied by a readily detectable lowering of thaw point. The magnitude of this change is indicative of the effectiveness of the treatment.

The crystals of milled soaps are oriented predominantly in the direction of extrusion, as can be demonstrated by examination of milled soap under polarized light. This is done by observing a thin section of the soap (the broad surfaces of which are substantially parallel with the direction of extrusion) placed between crossed Nicols, first with the section of soap normal to the direction of maximum light extinction, and then turned 45° from that position. The increase in light transmission in the second position is indicative of the orientation of the particles of the specimen and distinguishes milled and extruded soaps from framed soaps.

We have described above means of measuring certain milled soap characteristics. Among other such characteristics which are less readily susceptible to quantitative measurement are uniformity of texture, smooth wax-like feel, glossiness, tendency toward “cracking” under relatively mild conditions and tendency to return to substantially the original shape and appearance after drying, and freedom from warping during aging. These are characteristics which are easily recognizable when our product is compared with framed soaps of the same composition.

Most observations which are available as criteria for the existence or non-existence of characteristic milled soap properties are more easily applicable to bar soap than to soap in flake or other compound form. However, comparisons of rate of solutions, of transluence, and of firmness can also be made on flakes, and X-ray studies to determine phase and dilatometer runs to determine thaw point can be made on the soap irrespective of its form. We specifically contemplate soap possessing milled soap characteristics, but in particular forms such as flakes, granules, powders, lumps, threads, etc., as well as in the conventional milled soap bar form.

**ELECTROLYTE AND WATER CONTENT OF COMPOSITIONS OF THE INVENTION**

In contrast to the prior art, according to which milled soaps are prepared from soaps containing less than about 20% water and less than about 1% sodium chloride, our invention provides milled soaps of up to 35% or 40% water and of high salt content. The heart of our invention is in the relationship between water, salt and soap content, and our discovery of the range of ratios of those to one another within which range milled soaps can be made by our process. If, with high water content, the salt content is too low, a soft, opaque soap in delta phase is commonly obtained, which swells little and cracks badly when soaked in water, which is not readily soluble. If salt is too high, it lathers poorly as a result of the difficulty of removing soap from the wet bar by rubbing. If, on the other hand, the salt content is too high, a bar is obtained which is opaque, tends to crumble badly, has little power to pick up water or swell when soaked, is not readily soluble, is poor in lathering power, and is commonly predominantly omega. The limiting salt and water content of soaps which are capable of conversion to the milled soap type by our process will be discussed more explicitly hereinafter.

Adjusting the salt and water content of our soaps so that they are properly related, and subjected to the mechanical treatment described during extrusion, we find that a soap of a given composition can be milled into a product having a lower thaw point. The magnitude of this change is indicative of the effectiveness of the treatment.

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Most observations which are available as criteria for the existence or non-existence of characteristic milled soap properties are more easily applicable to bar soap than to soap in flake or other compound form. However, comparisons of rate of solutions, of transluence, and of firmness can also be made on flakes, and X-ray studies to determine phase and dilatometer runs to determine thaw point can be made on the soap irrespective of its form. We specifically contemplate soap possessing milled soap characteristics, but in particular forms such as flakes, granules, powders, lumps, threads, etc., as well as in the conventional milled soap bar form.
differing for different salts and for different soaps, but the amounts of any of the salts relative to each other being practically the same irrespective of the particular soap in question. In a somewhat analogous way, these salts affect those properties of high-moisture plastic solid soaps which determine whether or not milled soap characteristics are producible therein by a shearing and pressure treatment.

In order to practice our invention with soaps of from 20% to 40% water content, it is in general necessary to adjust the salt content to about 1% to 12% by weight, the remainder being substantially all soap. The various salts are of varied effectiveness with respect to milled soap properties; for each there is a range of ratios of salt:soap:water within which milled soap properties are producible by shearing treatment and pressure. These ranges can be defined by means of graphs in which the salt content is plotted as ordinate against the water content as abscissa. By subtracting the sum of the salt and water contents from 100%, the approximate per cent real soap is arrived at, subject to minor corrections for fractional percentages of glycerc, unsaponifiable matters, carbonated alkali, etc., which may be present.

Figs. 1, 2, 3, 4, and 5 are such graphs for five common salts, sodium chloride, penta-sodium tripolyphosphate, tetradsodium pyrophosphate, trisodium orthophosphate and sodium silicate. Fig. 5 is found generally applicable with sodium silicates having a SiO₂:Na₂O ratio of from about 2.5:1 to about 3.5:1. On each of these graphs, there is an area A within which our novel invention can be practiced. With higher salt or water contents, our invention can be practiced only with difficulty or not at all, the products of the shearing and pressure treatment being deficient in characterizing milled soap properties. By way of illustrating the use of these graphs, reference is made to Fig. 1. If it is desired to make a milled type of soap having a moisture content of, for example, 33%, the salt content should be adjusted to between 1.4% and 4.3%.

These graphs are intended to serve as guides for the processing of the typical soap under discussion, the optimum values in any particular case being readily determinable as judged by the previously mentioned product characteristics. Generally speaking, for any desired moisture content within the range of the invention suitable salt contents lie on and close to the straight line drawn from the origin (20% moisture, 1% salt) to the diagonally opposite corner of the area A, and salt contents further removed from this line should be employed with some caution.

**SUITABLE FAT STOCKS FOR SOAPS OF THE INVENTION**

While reference has been made hitherto to a sodium soap from 80% tallow and 20% coconut oil, and while this formula yields a high moisture bar of particularly desirable properties, formulae of about 70% to 85% tallow and about 15% to 30% coconut oil are well suited for such bars. Such soaps are preferably extruded or milled at about 105° to 125° F., and when given mechanical working and compacting at this temperature, they can be converted predominantly into the beta phase (i.e., more than 50% beta as determined by X-ray analysis), and typical milled soap properties can be imparted to them. With increasing amounts of coconut oil above about ½ of the fat formula, it becomes increasingly difficult to convert the soap into the milled soap type, although formulae of as high as 50% coconut oil can be employed. When the coconut oil percentage in the stock formula much exceeds 50%, the soaps thus produced are in many cases no longer predominantly in the beta phase or characterized by the described milled soap properties.

We are here using coconut oil as a specific example, but generally similar considerations apply when other oils of the foregoing type are substituted for coconut oil. By oils of the coconut oil group we mean to designate all vegetable seed oils or fats at least 50% by weight of the combined fatty acids of which are lauric and/or myristic acids. These oils (many examples of which are given in Hillich’s “The Chemical Constitution of Natural Fats,” second edition (1947), pages 198-205) are commonly derived from seed of members of the botanical families Lauraceae (tangkakalak kernel oil for example), Myristicaceae (teakuba nut oil for example), Vochysiacae (gabi oil), etc. (For example), Salvadareaceae (chakan kernel oil for example), Simarubaraceae (dika nut oil for example), and more especially the Palmae family. Coconut oil is the outstanding example of an oil derived from the seed of the Palmae family, but other non-limiting examples of such oils are oils of murumuru, icucuma, colhane, ourocouy, babassu and palm kernel.

The factor determining whether or not a given fat will yield a soap which can readily be converted predominantly into beta phase and which will have typical milled soap properties, is not the particular source of the fat, but rather the composition of the combined fatty acids therein. Thus while it is possible to practice our invention with soap from stock mixtures containing more than 50% by weight of some of the oils of the coconut oil group, we have in general experienced difficulty in accomplishing the objects of our invention when more than about 50% by weight of the fatty acids of the soap are saturated fatty acids of less than 16 carbon atoms. Tallow, grease and palm oil are examples of fats which, either alone or mixed with each other or with coconut oil, can be used for making the soaps of our invention. Highly unsaturated vegetable or marine oils are partially hydrogenated if they are to be used in any substantial proportion in the fat stock, since without hydrogenation their soap is too soft to be suitable for our treatment. They can, however, be mixed in limited proportions with other more highly saturated fat. In general, in order that our process be successful, the soap should be made of fats of more than 25 iodine value which contain not more than 40% saturated fatty acids of less than 16 carbon atoms and at least 20% by weight of saturated fatty acids of 16 to 22 carbon atoms.

As in the case of conventional milled soap manufacture, a limited substitution of potassium for sodium as the soap cation is permissible in the practice of our invention, so long as this substitution is not so great as to deprive the product of characteristic milled soap properties. In the same fashion potassium salts may be substituted for the soaps thus made. Similar considerations apply in the case of other soapmaking cations, such for example as the ammonium ion or substituted ammonium ions (e.g. triethanolamine soaps).
EXAMPLES

The following examples are illustrative of our process and of the detergent products made thereby, but it is to be understood that they are illustrative only and that our invention is not limited thereby but only by the terms of the appended claims. Examples have been selected in which the shearing forces and pressure were obtained by and during extrusion, such selection being made because of the more precise control and measurement of temperature, moisture content and pressure which are possible in extrusions. However, it will be understood that other means of applying shearing forces under pressure are feasible, such for example as milling and plodding. Unless otherwise noted, the extrusions of the examples were through a small cylindrical orifice, one from an, fused-steel chamber into another chamber, one wall of each chamber being movable and consisting of the face of a hydraulically operated piston. The two pistons move synchronously, but the pressure on each is independently controlled. Hence the soap is extruded from one chamber at high pressure into another chamber at a pressure which is lower but sufficient to mould the soap into a coherent bar suitable for stamping in conventional manner. When such soap is heated to the molten condition and then allowed to solidify by cooling to room temperature without agitation, it is referred to as "framed soap."

Example 1.—Soap was made by the conventional boiled, settled process from a stock formula of:

- 23% coconut oil
- 20.0% marine oil, hydrogenated to 70 iodine value
- 22.5% tallow
- 5.7% palm oil

To the molten kettle soap, which contained 0.4% sodium chlorite and about 30% water, there was added in a crutter enough sodium chlorite to bring the salt content to 2.75%. The hot mixture was allowed to cool to room temperature and solidify. It was then forced back and forth 34 times from one chamber into another through a slit orifice 1/4" x 3/4" in cross section. The pressure on the high pressure side of the orifice was 550 to 640 lbs./in.

Example 2.—Another lot of the molten kettle soap of Example 1 was partially dried and was adjusted in a crutter to a sodium chloride content of 1.5% and a water content of 22.2%. This soap was cooled in a frame and was then extruded under the same conditions as in Example 1 except that the pressure on the high side was from 675 to 840 lbs./in.

Example 3.—A mixture of 80% of mixed tallow and grease and 20% coconut oil was saponified with caustic soda according to the conventional boiled, settled process. The mixture of fats had an iodine value of 47.5. Saponified with 2.1% of NaCl, 214.3 and titer of 38. To the molten soap in a crutter, salt and water were added to adjust the mixture to 32.4% H2O and 3.8% NaCl. It was framed and then extruded 40 times at 90° to 100°F through 122 round orifices 3/8" in diameter. The resulting bar of soap was homogeneous, waxy and translucent in appearance, free-lathering, firm and in the beta phase.

Example 4.—Another lot of the kettle soap of Example 3 was partially dried and was then adjusted in the crutter to 26.4% H2O and 3.8% NaCl. It was then treated as in Example 3 except that the extrusion was at 120°F, at a pressure of 12,500 lbs./in.

Example 5.—Soap was made by the usual boiled, settled process from 80% mixed tallow and grease and 20% coconut oil, the mixed soap stocks having iodine value 42.6, saponification value 213.2 and titer 39.7. To the molten soap in a crutter, salt was added to bring the composition to 29.5% water content and 1.8% NaCl content. After framing and cooling, the soap was extruded through 122 orifices 3/8" in diameter. Extrusion was at 65 to 68°F, and was repeated 40 times. The result was a translucent bar of soap, smooth and waxy in feel and appearance, firm, free lathering, in beta phase and obviously a milled soap type.

Example 6.—Soap was made by the conventional boiled, settled process from a mixture of tallow, grease and coconut oil, the mixed soap stocks having an iodine value of 41.4, saponification value of 213.8 and titer 39.3. To the molten soap the usual amount was added to a water content of 31.2% and a salt content of 2.43%. It was framed and then extruded 40 times at 80 to 90°F through an orifice 3/8" in diameter, at a pressure of about 12,500 lbs./in.

The resulting soap was in the beta phase and possessed the translucency, firmness, free lathering ability, ready solubility...
in water, water-binding power and other properties characteristic of milled soap.

Example 7—Tallow kettle soap was mixed in the crucible with water and trioctanol orthophosphate and then allowed to cool and solidify to a final content of 33.5% water, 7.5% NaPOs and 0.58% NaCl. It was then extruded 40 times at 80° F. through a 1/4" orifice at a forward pressure of 50 lbs./in.² and a back pressure of 75 lbs./in.². The resulting bar was smooth, homogeneous, firm, translucent and readily soluble in water, and was in the beta phase.

Example 8—Tallow soap, made by the usual soap boiling method, was mixed in a crucible with commercial sodium silicate having a SiO₂:NaO ratio of 2.58:1. The crucible mixture was solidified by rapid cooling with slight separation of liquid and the solid soap was then extruded 40 times through a 3/8" orifice at 90° F. to form a bar, the forward pressure being 245 lbs./in.² and the back pressure in the chamber in which the bar of soap was molded being 100 lbs./in.². The resulting bar contained 8.5% silicate solids, 0.63% NaCl and 32.5% H₂O, was in beta phase and possessed characteristic milled soap properties.

Example 9.—The kettle soap of Example 8 was mixed in the crucible with tetrasodium pyrophosphate and additional sodium chloride and water. The crucible mixture was solidified rapidly by cooling, with only minor separation of liquid, and was then extruded 40 times through a 1/4" orifice at 110° F., the forward pressure being 105 lbs./in.² and the back pressure being 10 lbs./in.². The resulting bar contained 18% NaPO₄, 3.48% NaCl and 37.2% H₂O. It was in beta phase and possessed characteristic milled soap properties.

Example 10.—Another batch of the kettle soap of Example 1 was flash dried and then mixed in a crucible with tetrasodium pyrophosphate and water to a final composition of 5% NaPO₄, 0.5% NaCl and 22.2% H₂O. It was then framed and extruded 40 times through a slit orifice at a pressure of 490 to 660 lbs./in.² on the high side and 121 lbs./in.² on the low side, the temperature being 115° F. The resulting bar possessed typical milled soap properties.

Example 11.—Another lot of the molten kettle soap of Example 1 was mixed in a crucible with pentasodium tripolyphosphate to a composition containing 5.0% tripolyphosphate and 27.2% H₂O. It was cooled in a frame until solid and was then extruded 24 times at 100° F. through a slit orifice, from a high pressure of 565 to 604 lbs./in.² to a low pressure of 143 lbs./in.². The resulting bar of soap was in beta phase. It was translucent, firm (Mullen breaking test, 46 lbs./in.²), waxy, etc., being a typical milled soap in properties.

Example 12.—This example corresponds to Example 11 except that the final composition contained 10% sodium tripolyphosphate and 28.4% H₂O. The final soap was in beta phase and possessed the characteristic milled soap properties described in Example 11, although it was slightly softer than the soap of Example 11.

In the foregoing examples one can successfully employ milling and plodding instead of the repeated extrusions which we have described. Due precautions should be taken to provide adequate working of the soap and to avoid excessive moisture loss.

Having thus described our invention, what we claim and desire to secure by Letters Patent is: 1. In the process of preparing a detergent product of milled soap properties from a mixture of water, soap-compatible sodium salt possessing the power to "salt out" molten neat soap in the kettle, and a soap of a fat stock of iodine value above 50 not more than 40% of the combined fatty acids of which are saturated and of less than 16 carbon atoms and at least 20% of the combined fatty acids of which are saturated and of from 16 to 22 carbon atoms, the steps which comprise (a) adjusting the salt content of the mixture to from 1% to 12% and the water content to from 20% to 40% by weight, said contents being so proportioned that the soap properties are producible in the mixture by kneading, shearing and compacting treatment under pressure at a temperature below its solidification temperature, and (b) subjecting said adjusted mixture in the plastic solid condition to kneading, shearing and compacting treatment under pressure at a temperature within the range of about 80° to about 125° F. and such that the mixture is below its solidification temperature and below that at which neat soap can exist in stable equilibrium in the system but above the temperature at which it becomes brittle, whereby a product is produced the soap in which is waxy, translucent and predominantly in the beta phase.

2. The process of claim 1 wherein the salt is a member of the group consisting of sodium chloride, tetrasodium pyrophosphate, pentasodium orthophosphate, pentasodium tripolyphosphate, sodium silicate having a SiO₂:NaO ratio of from 2:1 to 4:1 and mixtures thereof.

3. The process of claim 1 wherein the salt is sodium chloride and the electrolyte and water contents lie within area A of Fig. 2.

4. The process of claim 1 wherein the salt is tetrasodium pyrophosphate and the electrolyte and water contents lie within area A of Fig. 2.

5. The process of claim 1 wherein the salt is pentasodium tripolyphosphate and the electrolyte and water contents lie within area A of Fig. 3.

6. The process of claim 1 wherein the salt is triosodium orthophosphate and the electrolyte and water contents lie within area A of Fig. 4.

7. The process of claim 1 wherein the salt is sodium silicate having a SiO₂:NaO ratio of from 2.5:1 to 5.1 and the electrolyte and water contents lie within area A of Fig. 5.

8. The process of claim 1 wherein the mixture is subjected to shearing forces under pressure at a temperature within the range in which the mixture is a plastic solid composed essentially of solid soap crystals and nigré.

9. The process of preparing toilet soap bars which comprises mechanically working and compacting a mixture of water, soap-compatible sodium salt possessing the power to "salt out" molten neat soap in the kettle, and soap of a fat stock of iodine value above 23 not more than 40% of the combined fatty acids of which are saturated and of less than 16 carbon atoms and at least 20% of the combined fatty acids of which are saturated and of from 16 to 22 carbon atoms, the concentration of water being from 20% to 40% and that of salt from 1% to 12% by weight, said contents being so proportioned that milled soap properties are producible in the mixture by mechanical working, kneading and compacting, said working, kneading and compacting being at a temperature of about 80° to 125° F. and such that the mixture is below its solidification point and is a plastic solid, and
17. continuing said working, kneading and compaction until the soap is waxy, translucent and homogeneous in all areas, especially in the beta phase, and has milled soap characteristics.

10. The process of preparing high-moisture milled type of toilet soap from a mixture of soap, water and sodium chloride, the soap being the sodium oleate of a meat stock of 15% to 30% coconut oil and about 85% to 70% tallow, which process comprises adjusting the water and salt content of said mixture to a point within area A of Fig. 1 hereof, milling said adjusted mixture in plastic solid condition at a temperature which is below its solidification point and is between about 100° to 125° F., and extruding the soap within said temperature range in continuous bar form through an orifice of restricted area, whereby a visually homogenized extruded bar of milled soap characteristics is produced, the soap therein being waxy, translucent and predominantly in the beta phase.

11. A high moisture detergent having milled soap characteristics and consisting essentially of soap which is predominantly sodium soap of a fat stock of iodine value above 25 at least 20% of the combined fatty acids of which are saturated and of from 16 to 22 carbon atoms and not more than 40% of the combined fatty acids of which are saturated and of less than 16 carbon atoms, soap-compatible sodium salt possessing the power to "soften" water, water content from 20% to 40% and the salt content being from 1% to 12% by weight thereof, soap constituting substantially all of the remainder of the detergent, the said soap being waxy, translucent and predominantly in the beta phase.

12. A high moisture detergent having milled soap characteristics and consisting essentially of soap, water, and at least one member of the group of salts consisting of sodium chloride, tetrasodium pyrophosphate, pentasodium tripolyphosphate, trisodium orthophosphate, and sodium silicate having a SiO₂:Na₂O ratio of from 2:1 to 4:1, the water content being from 20% to 40% and the salt content being from 1% to 12% by weight of the detergent, soap constituting substantially all of the remainder of the detergent, said soap being sodium soap of a fat stock of iodine value above 25, at least 20% of the combined fatty acids of which are saturated and of from 16 to 22 carbon atoms and not more than 40% of the combined fatty acids of which are saturated and of less than 16 carbon atoms, and sodium silicate having a SiO₂:Na₂O ratio of from 2:1 to 4:1, the water content being from 20% to 40% and the salt content being from 1% to 12% by weight of the detergent, soap constituting substantially all of the remainder of the detergent, the said soap being sodium soap of a fat stock of iodine value above 25, at least 20% of the combined fatty acids of which are saturated and of from 16 to 22 carbon atoms and not more than 40% of the combined fatty acids of which are saturated and of less than 16 carbon atoms.

13. A high moisture detergent having milled soap characteristics and consisting essentially of soap which is predominantly soap of a fat stock of iodine value above 25, at least 20% of the combined fatty acids of which are saturated and of from 16 to 22 carbon atoms and not more than 40% of the combined fatty acids of which are saturated and of less than 16 carbon atoms, and sodium silicate having a SiO₂:Na₂O ratio of from 2:1 to 4:1, the water content being from 20% to 40% and the salt content being from 1% to 12% by weight of the detergent, soap constituting substantially all of the remainder of the detergent, the said soap being sodium soap of a fat stock of iodine value above 25, at least 20% of the combined fatty acids of which are saturated and of from 16 to 22 carbon atoms and not more than 40% of the combined fatty acids of which are saturated and of less than 16 carbon atoms.

14. A high moisture detergent having milled soap characteristics and consisting essentially of soap which is predominantly soap of a fat stock of iodine value above 25 at least 20% of the combined fatty acids of which are saturated and of from 16 to 22 carbon atoms and not more than 40% of the combined fatty acids of which are saturated and of less than 16 carbon atoms, water and tetrasodium pyrophosphate, the water and tetrasodium pyrophosphate contents being such as to lie within area A of Fig. 2 hereof and soap constituting substantially all of the remainder of the detergent, the said soap being waxy, translucent and predominantly in the beta phase.

15. A high moisture detergent having milled soap characteristics and consisting essentially of soap which is predominantly soap of a fat stock of iodine value above 25 at least 20% of the combined fatty acids of which are saturated and of from 16 to 22 carbon atoms and not more than 40% of the combined fatty acids of which are saturated and of less than 16 carbon atoms, water and pentasodium tripolyphosphate, the water and pentasodium tripolyphosphate contents being such as to lie within area A of Fig. 3 hereof and soap constituting substantially all of the remainder of the detergent, the said soap being waxy, translucent and predominantly in the beta phase.

16. A high moisture detergent having milled soap characteristics and consisting essentially of soap which is predominantly soap of a fat stock of iodine value above 25 at least 20% of the combined fatty acids of which are saturated and of from 16 to 22 carbon atoms and not more than 40% of the combined fatty acids of which are saturated and of less than 16 carbon atoms, water and trisodium orthophosphate, the water and trisodium orthophosphate contents being such as to lie within area A of Fig. 4 hereof and soap constituting substantially all of the remainder of the detergent, the said soap being waxy, translucent and predominantly in the beta phase.

17. A high moisture detergent having milled soap characteristics and consisting essentially of soap which is predominantly soap of a fat stock of iodine value above 25 at least 20% of the combined fatty acids of which are saturated and of from 16 to 22 carbon atoms and not more than 40% of the combined fatty acids of which are saturated and of less than 16 carbon atoms, water and sodium silicate having a SiO₂:Na₂O ratio of from 2:1 to 4:1, the water content being from 20% to 40% and the salt content being from 1% to 12% by weight of the detergent, soap constituting substantially all of the remainder of the detergent, the said soap being sodium soap of a fat stock of iodine value above 25, at least 20% of the combined fatty acids of which are saturated and of from 16 to 22 carbon atoms and not more than 40% of the combined fatty acids of which are saturated and of less than 16 carbon atoms.

18. A high moisture toilet soap bar of milled type consisting essentially of a mixture of soap, water and sodium chloride, the soap being the sodium soap of a mixture of about 15% to 30% coconut oil and about 85% to 70% tallow, the salt and water content of said bar lying within area A of Fig. 1 hereof and soap constituting substantially all of the remainder of the detergent and being predominantly in the beta phase, the said bar being of the milled soap type and being firmer and more translucent than is the same bar after being melted and resolidified without agitation and the soap in said bar being predominantly in the beta phase.

19. In the process of preparing a detergent product of milled soap properties from a mixture of water, a soap-compatible sodium salt selected from the group consisting of sodium chloride, tetrasodium pyrophosphate, pentasodium tripolyphosphate, sodium silicate having a SiO₂:Na₂O ratio of from 2:1 to
4:1 and soap which is predominantly sodium soap of a fat stock of iodine value above 25 at least 20% of the combined fatty acids of which are saturated and of from 16 to 22 carbon atoms and not more than 40% of the combined fatty acids of which are saturated and of less than 16 carbon atoms, the steps which comprise (a) adjusting the salt content of the mixture between 1 and 12% and the water content between 20 and 40% by weight so as to produce a mixture the composition of which lies close to a straight line extending from the origin represented by 20% moisture, 1% salt to the diagonally opposite corner of area A of that one of the graphs in Figures 1–5 which applies to the particular salt selected, and (b) subjecting said adjusted mixture in the plastic solid condition to kneading, shearing and compacting treatment under pressure at a temperature below its solidification temperature and in the range of substantially 80° to substantially 120° F., whereby a product is produced the soap in which is waxy, translucent and predominantly in the beta phase.

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