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2,465,346

DEDUSTING PROCESS AND PRODUCT
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This invention relates to improvements in the manufacture of soaps and more particularly to powdered, granulated or comminuted soaps substantially devoid of objectionable finely divided soap dust particles; the invention relates also to methods of manufacturing such soaps.

Granulated or powdered soaps usually are formed in one of three well-known ways, namely, grinding, in which dried soap is comminuted and reduced to a powdered condition; spray-cooling, wherein a hot liquid soap that is capable of crystallizing is sprayed into a current of cooling air; and spray-drying, wherein a hot liquid or semi-liquid soap or soap mix is sprayed into a current of heated drying gas to evaporate a portion of the water and cause the soap to solidify as particles, or wherein soap is heated under pressure and flashed into cool air at lower pressure. Spray dried soaps may be either hollow or porous fragile particles.

Soaps of these types are usually packaged in cardboard cartons and are sold to the consuming public in this convenient form. While such soaps are highly desirable from the standpoint of ease in solubility and convenience in use, when they reach the consumer they contain a quantity of finely divided soap particles. A material proportion of this dust or fines is capable of being air borne. The portion of the dust capable of being air borne is usually released upon opening the carton and pouring the soap therefrom. It is this air borne soap dust that has been found in many cases to irritate the membranes of the nose and throat, causing coughing and sneezing.

The dust particles or fines also have a tendency to lump together on the surface of the water and resist solution. This characteristic of comminuted or powdered soap products has long been recognized in the industry and attempts have been made to eliminate the dust or at least materially reduce the quantity in the product.

The attempts to overcome this difficulty have led to improvements in spray-drying processes, by means of which the soap is formed into more or less rounded and discrete individual granules. Such granules are small enough to dissolve readily, but also large enough to minimize greatly the disadvantages of the so-called "dusting." The factors affecting the size and character of the soap granules produced in a spray-drying process are well known. For example, fine particles can be puffed to above dust size if high temperatures and other conditions are employed. However, large puffing generally is not desirable, for in doing this to the fine particles, the total product

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is bulked to such a large degree that in general there is present too small an amount of soap in any given volume, and the product does not have desirable sudsing properties. Furthermore, large puffing creates thin walled particles more susceptible to breakage, thus tending to increase the dust content of the product.

In spite of the exact control now possible, there is still generally an objectionable amount of dusty material in the product at the time the customer uses it. A proportion of "fines" unavoidably results from the commercial manufacturing processes. Dust formed during the process of manufacture could be made relatively unobjectionable in the final product by an efficient dedusting process after manufacture and prior to packaging. One of the principal causes of dust in the packaged product is the disintegration of the particles after the product is packaged. This may be because of conditions of storing and handling before or after the carton or package is opened for use.

Attempts to remove finely divided material of dust size by screening before packing have not been successful, however, for the reason that screens of sufficient fineness to allow separation of the dust become clogged, and, at best, remove only a part of the dust. Even assuming that a screening process removes the dust formed during the production of the soap, it cannot remove the dust which is formed in the package.

The formation of dust in the package results, in part, from disintegration of the soap particles during handling and is increased by drying out of the soap. A soap product such as, for example, a spray-dried soap, may consist of more or less spherical globules, each having one or more voids therein. A moisture content of between 10 percent to 20 percent is normally present in typical forms of granulated soaps at the time they are manufactured and packaged. This moisture content is sufficient to render the particles somewhat plastic and resistant to shattering. As long as this moisture content is maintained at substantially its initial amount, there is little tendency for the soap to form dust. However, if the soap dries, as is often the case, the plasticity of the particles is then reduced. Upon being subjected to shock or abrasion, the dried and friable particles will break or shatter and thus form additional quantities of dust. Low moisture content also impedes any tendency towards a reaggregation of the fines into larger agglomerates. In addition, loss of moisture permits a migration of fillers or builders in the soap, such as sodium

carbonate, sodium sulfate and sodium silicate, toward the surfaces of the particles where they deposit as crust or groups of crystals. These crystals are separable by abrasion from the larger particles as dust. In the case of the more alkaline fillers, such as sodium carbonate or sodium silicate, this form of dust is of a particularly irritating nature. There is also a tendency for the dust to remain in suspension in the air because of the presence of static charges of like sign on the particles. Such electrical dispersing charges may be created as a result of friction between the particles and the package during handling. The presence of adequate moisture content in the particles, however, largely eliminates the effects of static electricity.

The breaking up or disintegration of the soap particles also has the undesirable effect of reducing the apparent volume of the soap in the cartons. This creates an adverse psychological effect upon the user when the package of soap is opened, inasmuch as it would appear as if less than the full quantity of soap were present in the package.

It has been suggested that drying out of the soap particles might be avoided by packaging the granulated soap in moisture-proof containers. While this increases the cost of packaging, it will, of course, prevent drying of the soap as long as the package is unopened, but inasmuch as the entire contents of the soap package ordinarily is not used immediately after opening the package, the remainder of the soap will dry out readily upon standing in a dry place, and objectionable dust will be formed.

My invention may be utilized to overcome and avoid at least some, and preferably all, of the disadvantages and difficulties of the type indicated above, as well as others, in comminuted friable substances, and for preserving and improving such substances, particularly those of the type of soap. In this connection the invention may preferably be utilized to substantially reduce the tendency of granulated or powdered soaps or other such friable substances to disintegrate or change their physical state with consequent dust formation. The invention may also be preferably utilized to maintain the moisture content of soap particles substantially constant despite variations in relative humidity so as to minimize and prevent the drying out of the soap particles with a resultant undesirable formation of dust. The invention may be utilized to minimize or eliminate dust particles present in a soap product, for example, by maintaining the soap in an undried form so as to retain the strength of the soap particles; consequently there is little or no tendency for the soap particles to disintegrate or change their state. Under some circumstances the surface of the larger particles may hold the fines attached thereto or the fines may be held together through a cohesive action.

I have subsequently set forth preferred examples and illustrations of how my process may preferably be practiced to produce desirable products.

In accordance with the present invention the individual soap particles or granules, after they are formed, may be coated with a material that functions primarily because of its hygroscopic nature and which normally withdraws moisture from the atmosphere and thus prevents a migration or diffusion of the moisture from the interior of the soap particles. The hygroscopic

material in many cases also may act to bind dust particles to each other or to the larger particles. These dust particles may be present in the soap initially or may be formed if the soap is subjected to unusual conditions.

Of the materials which have been used in treating granulated soap, the polyhydric alcohols, are desirable compounds to use as coating materials to eliminate dust. These compounds assist in maintaining a definite water concentration in the soap inasmuch as under normal humidity conditions they will withdraw moisture from the atmosphere. This moisture will compensate for any that is lost by evaporation, or if more is withdrawn it will penetrate into the soap particles and improve their plasticity. Even under dry conditions moisture will only diffuse from the soap until a balance is reached between the atmospheric humidity and the ability of the polyhydric alcohol to withdraw moisture from the air. Under conditions such that the relative humidity is as low as 30 percent, it has been found that polyhydric alcohols, such as glycerin, ethylene glycol, diethylene glycol, sorbitol and mannitol, are satisfactory for preventing the dusting of the soap.

The organic and inorganic phosphates, such as, for example, dipotassium phosphate or potassium methyl or ethyl phosphate, also are satisfactory hygroscopic coating materials. Dipotassium phosphate is very effective at relative humidities in excess of about 30 percent. For example at 50 percent relative humidity, dipotassium phosphate will carry about 40 percent of water, which is adequate to maintain it in the form of an oily or mobile liquid. However, below about 30 percent relative humidity, it will dry out to a crystalline solid. Therefore, it is less suitable for use in treating soap which will be exposed to very low humidities. The presence of an alkyl group, such as an ethyl or methyl group, in place of one of the alkali metal ions, results in compounds which will retain their fluidity at extremely low humidities. Alkyl phosphates, having the formula $MRHPO_4$, in which M is an alkali metal and R is an alkyl group, therefore, are particularly suitable for use as coating agents for granulated soap under all conditions of relative humidity.

The above mentioned compounds also cause any dust particles to aggregate into larger particles or adhere to large soap granules. They also minimize the formation of static charges and thus reduce the tendency of any small soap particles to remain in suspension.

Other hygroscopic materials may be used such as potassium carbonate, potassium silicate, disodium phosphate and calcium or magnesium chloride. Other hygroscopic materials which also function as wetting agents may be used since these materials contribute to the plasticity of the soap particles because of their wetting nature and strengthen the bond between aggregates of fines and between the fines and the particles of normal size.

Hygroscopic substances or those which themselves may be conductors have the additional characteristic of preventing the formation of static charges on the soap particles for the reason that they render the surfaces of the particles conductive, and thus reduce the tendency of the dust to remain in suspension in air. Such substances which might be used for this purpose would include, for example, glycerin, dipotassium phosphate, sodium potassium ethyl

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phosphate, triethanolamine ethyl phosphate, glycerin and dipotassium phosphate, glycerin and sodium potassium ethyl phosphate, glycerin and potash coconut oil soap, water-soluble sulfonated oils and lithium chloride.

The various materials mentioned may be used alone or in any admixture.

These coating compositions should be present in sufficient proportion to obtain the desired results and yet not in such proportion that they will cause lumping and formation of aggregates of such size that the characteristic of rapid dissolving, which is desirable in granulated soaps, is materially reduced.

It is desirable to prevent excess quantities of the coating material from impinging upon the soap particles in order to prevent them from sticking or lumping together and interfering with their normally free-flowing properties. Since the coating material is applied only to the surface of the particles, very small quantities are required. This makes the process particularly economical and achieves results that would be prohibitive from the standpoint of cost if the material were incorporated within the soap before it was placed in granular form.

The amount of the hygroscopic material to be employed will depend upon the hygroscopic character of the material selected, the nature of the soap, the climatic conditions where the product is to be used, etc. Soaps made from certain fatty acids and alkalies and having certain fillers and builders have different degrees of natural plasticities and water retentivity. This fact coupled with the different hygroscopic character of various materials makes it difficult to state any range applicable to all of the hygroscopic materials. When glycerin is used, for example, an amount of about 0.5% to 6.0% based on the weight of the soap, is preferable. It is believed that in view of the generic disclosure and the specific example, one skilled in the art would readily be able to determine the amount to be used in any particular instance, depending upon the particular soap to be treated and the use to which it is to be put.

It will be understood that a greater or less amount of the coating composition referred to may be used, depending on the character and physical characteristics of the granulated soap treated. Greater amounts of these coating compositions may be used, although this increases the cost of treating the soap.

An embodiment of my invention that is of particular importance is the use of calcium chloride as a hygroscopic agent. Normally the addition of a calcium compound to soap or to water in which soap is to be used would be thought of as objectionable because it is the calcium which causes hardness in water. This is normally thought to be a disadvantage and calcium is an ingredient sought to be eliminated. It is, nevertheless, surprising that the amount of calcium chloride to be added to the surface of the soap particles as a hygroscopic material to preserve the moisture content may be so small as not to interfere seriously with the increase in hardness properties. For example, the amount of calcium chloride to be applied to the soap may be within the range of 0.25% to 1.0% based upon the weight of the soap. When this soap is used in water in a concentration of 0.4%, which is an ordinary concentration for laundry, the amount of calcium introduced into the wash water as a result of using the treated soap will increase the hardness

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of water only about 16 parts per million. This is very small considering the fact that hard water ordinarily runs as high as 100 to 500 parts per million in the hard water districts. The percentage increase is relatively small.

When the soap is made with tetrasodium pyrophosphate as a builder, it is desirable to have an amount of calcium present in the water as this increases the detergent properties. In soft water districts if the amount of calcium in the water is not sufficient it may be added to secure this effect. In accordance with this invention calcium chloride may be added to the soap not only to prevent dusting but also as a convenient way of introducing the necessary calcium into the wash water.

It is characteristic of the invention that the hygroscopic material is applied to the surface of the particles as distinguished from an ingredient that is admixed with the soap composition before it is dried. In the latter case, the hygroscopic material would not have an opportunity to exert its hygroscopic character to any marked extent. As the soap particle dries, the moisture first leaves the outer surface and it is the outer surface which dries first. The moisture from the interior then migrates through the particle to the outer surface. Thus if the outer surface can be kept moist as a result of the hygroscopic material, there will be no tendency for the normal moisture on the interior to leave the particles. Furthermore, since it is only the surface of the particle which is in contact with the air from which moisture could be absorbed by hygroscopicity, there is no point in having the hygroscopic material at any point other than on the outside surface of the soap particles. The hygroscopic material may be applied more or less uniformly over the surface of the soap particles and it may be applied more or less spotted or continuously over the surface. Since the hygroscopic agents will, for the greater part, be water-soluble, there is no objection to a continuous coating on the surface of the particles, although a partially spotted coating may be sufficient to keep the soap particles plastic with the coating of this amount and nature.

The hygroscopic material may be soluble in the soap or not. If not, it has the advantage that it will not tend to diffuse into the soap particles and will not require such a large quantity of material. On the other hand, the materials which penetrate tend to keep the entire mass of the soap particle plastic.

The coating materials may be applied to soap particles in various ways, and at any convenient time between the formation of the soap particles and the packaging step. The coating materials are preferably atomized and the soap particles allowed to fall through the atomized coating material. It is desirable to maintain the finest possible mist or fog of the material to be applied so that the most effective application can be made and even the finest of the dust particles forced into contact with the treating material. It has been found that a series of repeating treatments is effective in obtaining a desirable dust-free product. Also, the coating composition may be applied by spraying in a rotary drum through which the soap particles are tumbled.

The major portion of the soap particles treated will, for the most part, vary in particle size from about 20 to 80 mesh. They may be coated completely or partially with a single coating composition or a blend, simultaneously or in separate

stages, as already described. Any fine, objectionable, dust-like material will be coated and will tend to bond with the larger particles or with other fine material to form aggregates of a size larger than the average size of the dust particles. This will be the case not only with disintegrated or fine soap particles, but also with any dust formed as a result of the migration or separation of the ingredients ordinarily present in the soap composition, such as the hydrating salts.

It has been found that in general dust particles of a mesh of 140 or smaller are undesirable in the final product and are considered as dust. It has been found that dust particles of about 400 mesh or smaller, as calculated on the bases of formulas for standard screens, will be air borne under conditions of relatively low air movement. These air borne particles might average 50 microns in size and smaller. They are, therefore, particularly undesirable as they can readily move into contact with the membrane of the nasal passages to cause irritation. Larger particles than 400 mesh may be air borne under conditions of increased air movement and for the same reason should be rendered innocuous.

From the foregoing it will be apparent that the present invention has resulted in the production of a novel dustless type of granular or comminuted soap product and that a simple process has been provided for obtaining such dustless soap granules. It will be understood that the present process can be applied to any of the usual types of granulated or powdered soaps in order to overcome their objectionable dusting characteristics and that by means of the process shrinkage in packaging or in weight may be largely avoided.

This application is a continuation-in-part of my pending application Serial No. 351,111, filed August 3, 1940, now Patent No. 2,329,694.

It will be understood that coating compositions similar to but other than those mentioned specifically above may be used without departing from the invention. Therefore, the forms of the invention described above should be considered as illustrative and not as limiting the scope of the following claims.

I claim:

1. A process of treating granulated water soluble soap to prevent substantially the formation of dust therein, comprising applying to the particles of soap a superficial coating of a hygroscopic alkaline earth metal chloride in an amount to inhibit dehydration of the soap and the forma-

tion of soap dust particles, but not to increase greatly the hardness properties of the water when the soap is used in ordinary concentrations for laundry.

2. The process of claim 1 in which the soap particles are spray dried, and the alkaline earth metal chloride which is applied is calcium chloride.

3. The process of claim 2 in which the calcium chloride is applied to the surface of the soap particles by spraying.

4. The process of claim 3 in which the amount of calcium chloride applied is within the range of 0.25% to 1%, based on the weight of the soap.

5. A water soluble soap product comprising granulated soap particles having on at least a portion of their surfaces a superficial coating of a hygroscopic alkaline earth metal chloride in an amount to inhibit dehydration of the soap particles and dusting of the soap product, but not to increase greatly the hardness properties of the water when the soap product is used in ordinary concentrations in laundry.

6. The product of claim 5 in which the granulated soap particles are spray dried.

7. The product of claim 6 in which the alkaline earth metal chloride is calcium chloride.

8. The product of claim 7 in which the amount of calcium chloride is 0.25% to 1%.

9. The product of claim 8 in which the soap particles contain tetrasodium pyrophosphate as a builder.

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The following references are of record in the file of this patent:

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Certificate of Correction

Patent No. 2,465,346.

March 29, 1949.

JOHN W. BODMAN

It is hereby certified that errors appear in the printed specification of the above numbered patent requiring correction as follows:

Column 2, line 24, for "packing" read *packaging*; column 8, line 44, list of references cited, before the patent number "2,093,927" insert the following—

2,030,583 Haas Feb. 11, 1936

and that the said Letters Patent should be read with these corrections therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 4th day of October, A. D. 1949.

[SEAL]

THOMAS F. MURPHY,
Assistant Commissioner of Patents.