

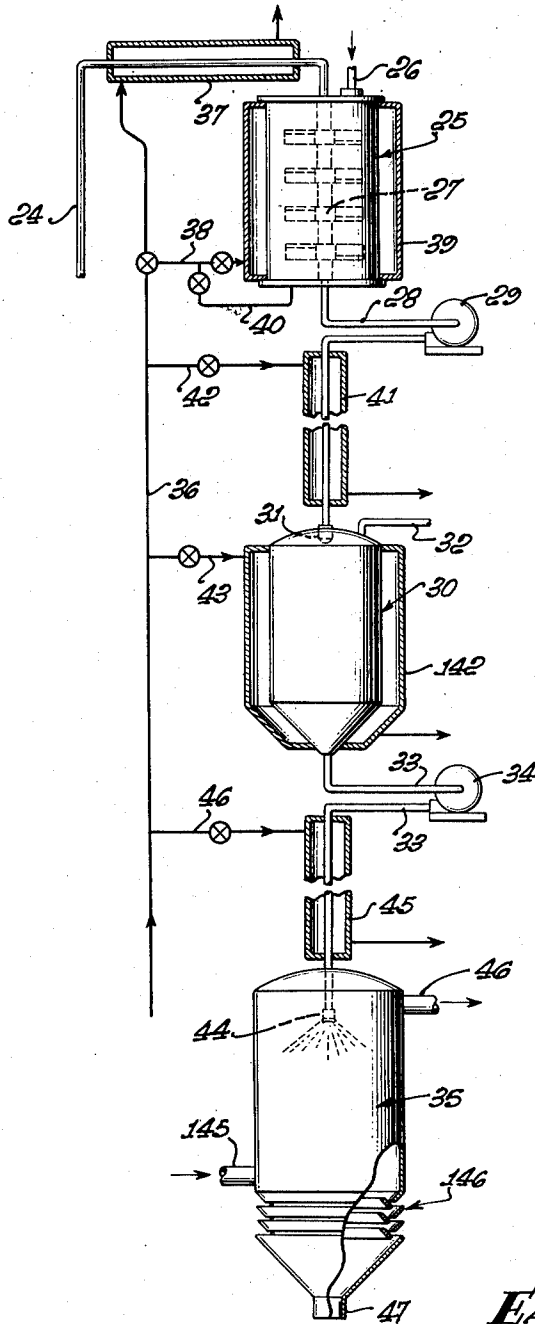
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SYSTEM FOR PRODUCING HIGH DENSITY DETERGENTS

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## SYSTEM FOR PRODUCING HIGH DENSITY DETERGENTS

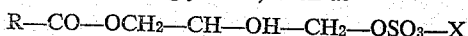
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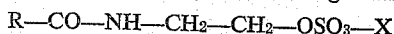
1 Claim. (Cl. 159-48)

This invention has to do generally with the manufacture of detergents suitable for use as washing compounds, and particularly with the production of such compounded detergents by methods having for their major object to give to the final and marketable products desirable solubility properties and high bulk densities. While applicable broadly to the manufacture of detergent compositions in general and whose densities are subject to increase by deaeration of a dryable slurry containing the detergent, the invention will be described typically, though as illustrative only, with reference to detergents containing as active ingredients the alkali metal salts of organic sulfonates and sulfates having such molecular structures as to possess detergent properties.

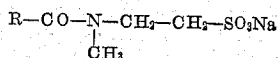
The active ingredient may be any of various sulfonated or sulfated compositions of the following class: alkali metal (e. g. sodium) alkyl aromatic sulfonates such as the mono alkyl benzenes described in the Lewis Patents Nos. 2,477,382 and 2,477,383, and also the alkylated naphthalene sulfonates, such as dibutyl naphthalene sulfonate, in all of which compounds the benzene or naphthalene radical is bonded to an alkyl group containing from 8 to 18 carbon atoms; alkyl sulfonate salts of the general formula  $R-SO_3-X$  in which R (as in the formulas below) is an alkyl group containing 8 to 18 carbon atoms and X is an alkali metal such as sodium, for example lauryl sulfonate and cetyl sulfonate; alkyl hydrogen sulfate salts of the general formula  $R-OSO_3-X$ , e. g. sodium oleyl sulfate and sodium lauryl sulfate; sulfated monoglycerides (of the type derived from coconut fatty acids and glycerine) such as



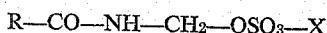
sulfated alkylolamides of fatty acids of the general formula



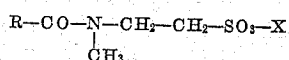
or



sulfated amides of the formula



or sulfonated amides



(e. g. the product sold under the trade name "Igepon-T"); sulfonated esters (such as "Igepon-A") of the general formula  $R-CO-OCH_2-CH_2-SO_3-X$ . To any or mixtures of the foregoing may be added any of the suitable alkylolamides in which the amide radical contains from 8 to 18 carbon atoms.

Suitable builders include the usual salts including sodium sulfate, sodium carbonate, alkali metal silicates and any of various polyphosphates.

It has been customary in the manufacture of such detergents to produce first an aqueous slurry of the alkali metal organic sulfonates or sulfates, to which are added

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one or more salts or additives such as soda ash, sodium bicarbonate, alkali metal silicate, borax, sodium sulfate, sodium chloride, fuller's earth and the like, to produce a mixture or slurry dryable to any final form, e. g. as by spray drying, drum drying, or tray drying. Considering spray drying as typical, it is desirable that the solid granules resulting from spray drying of the slurry, have a high bulk density, say upward of 0.40 or above. Now by the usual methods of slurry treatment and dehydration, potentially high bulk densities of final product either are not obtainable, or can be reached only as a result of relatively inconvenient and prolonged procedures.

The use of packaged soaps and detergents, rather than bar soaps, for both dish washing and the washing of cottons has progressed steadily during recent years. Both drum dried and spray dried types of product are packaged, with the preference for household use going to spray dried products because of their quicker solubility. One major limitation of such products, especially the spray dried detergents, is that their densities are not high enough for the product to have maximum usefulness. It is desirable that at least as much weight of the spray dried product be put into a box as is possible with granulated soap, but this objective cannot be attained by conventional methods. In accordance with the process disclosed in my Patent No. 2,606,156, issued August 5, 1952, on "Deaeration and Drying of Water Soluble Sulfonated Detergent Compositions," an important step toward achieving increased densities in the dried detergent product is accomplished by deaeration of the slurry preparatory to drying, as by passing the slurry from the mixer through a zone maintained under subatmospheric pressure, typically 5 to 250 mm. absolute that may be regulated in accordance with the degree of deaeration desired.

A general object of the present invention is to provide a new system for combined heating and deaeration of the slurry that affords improved density control and even greater ultimate densities in the final drying, and especially in a spray type drying operation. Heating alone has previously been tried, but found unsuccessful to any important degree, as a method for deaerating the slurry. The major and controlling difficulty found was that the consistency of mixes after the incorporation of the dry additives with the wet sulfonate, is such that even under heat, the dissolved or occluded air does not leave the mix. Continuance of the heating in order to favor eventual removal of air is not feasible because the presence of the detergent active ingredients causes a steady thickening and gellation of the entire mass. Accordingly, the thickening and gelling effect has led to abandonment of heating alone as a method of deaerating the slurry. I have now discovered that the efficiency of vacuum deaeration can be improved and regions of density of the dried products otherwise unobtainable can be reached by controlled heating of the slurry in such relationships to the vacuum deaeration stage as will later be explained in detail.

The system may employ apparatus comprising a mixing chamber or zone in which an aqueous sulfonate slurry stream is thoroughly admixed with the solid additives or builders, and an evacuated deaerating zone or chamber receiving the slurry from the mixing zone and within which the slurry is subjected to division and extended particle or surface exposure, typically by spraying the slurry within an atmosphere maintained in the aforesaid 5 to 250 mm. absolute pressure range, at which a considerable and even major portion of the air leaves the slurry. The deaeration is accompanied by water vaporization from the slurry, with the consequent tendency for the slurry to cool in proportion to the degree of deaera-

tion and water vaporization. From the deaerating chamber the slurry is pumped to the drier, preferably through spray nozzles which disperse the deaerated slurry in small droplets within the heated atmosphere of the drier where in final drying of the slurry occurs at the pressure at which spray driers ordinarily are operated, that is, at about atmospheric pressure.

It is preferred that the aerated slurry going to the vacuum chamber have a temperature in the order of about 110 to 160° F. in order to compensate for the tendency of the slurry to cool in the deaerator and thus preclude the possibility of excessive thickening of the slurry during its deaeration. In the mixing of some detergents and builders, as for example those having higher phosphate contents, sufficient heat of solution may be evolved as to raise the slurry temperature to within the desired temperature range. Where this is not the case, provision may be made for heating the sulfonate slurry in advance of the mixing zone, or the sulfonate and builder mixture within the mixing zone, or between the mixing and deaerating zones, so that the slurry will enter the deaerating zone at a temperature within about the 110 to 160° F. range. In general it is found desirable or necessary to bring the slurry up to the desired temperature within this range, within a period of time sufficiently short to avoid any consequential consistency increase or gellation of the slurry due to heating. While the temperature of serious gellation of any given slurry will be dependent upon its particular composition, it may be said that in general the slurry temperature rise should occur within a period of time not to exceed about 30 minutes.

It is found that the degree and effectiveness of deaeration of the slurry in the evacuated zone are materially increased by reason of the preheated condition. As an illustrative comparison, it has been observed that a slurry entering the deaerating zone (absolute pressure about 50 mm. of mercury) at a temperature of 150° F., lost twice the amount of air lost by the same slurry at a temperature around 110° F. passed through the deaerator under corresponding pressure.

With no further differences, the combined heating and deaeration contemplated by the invention gives a finished dried product that is approximately 10% denser than it would be where the slurry temperature during deaeration was kept relatively low, e. g., in the neighborhood of 100° F.

The invention has a further and particularly important concept in providing for heating of the slurry after its deaeration and with consequent temperature and fluidity preconditioning that result in still higher densities of the dried product. As will be understood, where the slurry is not heated in advance of or within the deaerator, subsequent heating may compensate for any cooling of the slurry in the deaerator. In any event, heating of the slurry during its flow from the deaerator to the drier within certain temperature limits directly favors the desirable increased density of the product for a number of reasons, including, (1) fluidizing or lowering the viscosity of the slurry ahead of the drier spray nozzle, with consequent lowering of the required nozzle pressure, the effect of which is to maintain larger average sprayed particle size in the drier and therefore less dust in the product, (2) the number of pounds per hour of slurry that can be spray dried is increased, (3) deaeration together with the heating is particularly beneficial in that heating of a slurry that has not been deaerated results in a puffy and non-uniform spray at the nozzle and consequent loss of particle size control, and (4) vacuum deaeration and heating permit a temperature and time control which can be utilized to permit the maximum effective heat input to the slurry without exceeding the critical temperature and time conditions that will result in its gellation. As illustrative of specific heating conditions, it is found that the advantages of slurry heating immediately in advance of the spray drier may be realized by heating a stream of

the deaerated slurry being pumped from the evacuated chamber to the drier nozzles to a temperature in the region of 120 to 190° F. over a controlled period of between 5 to 100 seconds.

Heating of the slurry within or beyond the deaerating zone produces a fluidizing effect of which advantage is taken along with a condition of at least practical similarity which is found to result from deaeration of the slurry. It is found that the fluidity, or at least the pumpability of the deaerated slurry, all other conditions being equal, is greater or better than that of the same slurry not deaerated. While the advantages of increased fluidity or better pumpability of the deaerated slurry might be lost entirely or in part by excessive cooling in the deaerator, provision for heating the deaerated slurry assures maintenance and utilization of fluidizing effects and advantages of both deaeration and heating.

In certain types of spray drying towers slurries may be dried within density limits which are suitable as low density products, without either deaeration or heating of the feed. If these slurries are deaerated in the present type of spray drying tower the densities are too high for light duty products, and heating within certain temperature limits will further increase the density outside of desirable limits. However, if the combination of heating and deaerating is such that the feed temperature is about 240° F. the density is then reduced to a point suitable for light duty products. This accomplishes, then, the production of the desired product at a much increased tower capacity.

All the features and objects of the invention, as well as the structural and method details of a typical commercially operable system, will be more fully understood from the following detailed description of the accompanying drawing, wherein the system is illustrated diagrammatically and in flow sheet form.

A neutralized slurry, typically an aqueous sodium alkyl aryl sulfonate slurry, is delivered through line 24 for delivery to the mixer 25. Suitable solid builders or fillers are added through inlet 26 to the slurry in the mixer and the components are subjected to thorough mixing as by a mechanically driven agitator diagrammatically indicated at 27. From typical operating data, it is found that the specified gravity of the neutralized slurry going to the mixer may be within the range of about 0.84 to 1.14. The sulfonated slurry after compounding with the additives in the mixer and containing for example 8 to 50% by weight of the neutralized active detergent, has an increased density within the range of about 1.15 to 1.20. This slurry then is continuously withdrawn from the mixer through line 28 and discharged by pump 29 into a deaerating chamber 30 preferably in finely divided or sprayed form, as through a spray nozzle 31 in the head of the chamber. The latter is connected through line 32 with a suitable evacuator, so that the deaerator is operated under a pressure considerably below atmospheric pressure, and typically within the range of 5 to 250 mm. absolute. Ordinarily the temperature of the compounded slurry will be approximately atmospheric temperature. Being dispersed in finely divided form within the evacuated zone, the compounded slurry is effectively depleted of its air content, as well as some moisture, in accordance with the pressure existing in the deaerator, and its density is brought within the range of say 1.20 to 1.44.

The deaerated slurry is continuously withdrawn from the deaerator through line 33 and is discharged by pump 34 into a suitable drier 35, preferably of the spray type within which the slurry is reduced to finely divided form and dried by contact with hot gases or air, resulting in the production of the detergent as granulated particles. As previously indicated, the primary effect and advantage of the deaeration is the production of granules having higher bulk densities, this property apparently being due to the fact that whereas ordinarily the presence of

air in the slurry being spray dried tends to remain entrapped and to expand therein, thus enlarging the hollow globular particles and thinning the shells, preliminary removal of the air in accordance with the invention enables the particles to dry in more compact and dense form, free from excessive air expansion.

The present invention is concerned primarily with the effects and advantages gained by combined heating and deaeration of the slurry. To that end, provision may be made for heating the slurry in any convenient manner, at the locations where heating is required in accordance with particular conditions involved in a given instance.

Temperature conditioning of the slurry in the mixer by heat from extraneous source, when required, may be effected by steam taken from line 36 to a jacket 37 about line 24 in advance of the mixer 25, or the slurry may be heated directly in the mixer as by steam supplied through line 38 to the mixer jacket 39, or by direct injection through line 40 into the slurry mass in the mixer. Heating in advance of or within the mixer may in some instances be substituted by heating the slurry stream between the mixer and the deaerator 30, as by means of a jacket 41 about line 28 and supplied with steam through line 42.

Where it is desired to heat the slurry while it undergoes deaeration, the chamber 30 may be enclosed within a jacket 142 to which steam is fed through line 43.

A particularly important provision for heating the deaerated slurry stream between the deaerator and spray tower, and preferably while the stream is being continuously discharged to the drier spray nozzle or nozzles 44, may be served by jacketing at 45 the line 33 throughout a sufficient length of line to raise the slurry temperature to the proper limit within a period of time sufficiently short to prevent gellation in advance of the nozzle 44. Steam is supplied to the jacket 35 through line 46.

The showing at 35 is to be regarded as diagrammatically illustrative of any suitable type of spray drier within which the slurry being discharged from the spray nozzle is dried as by heated air circulated through the ducts 145 and 146, the finally granulated dried product after passing through the atmospheric cooling section 46 of the drier being discharged through outlet 47.

To cite a typical batching procedure involved in making a low density light duty detergent, a slurry containing 4391 lbs. of water, 1636 lbs. of sodium sulfate and 2195 lbs. of sodium alkyl sulfonate was pumped into the mixing tank. The temperature of this slurry was brought to 100-105° F. by injecting live steam up through the mix tank. To the slurry was added 500 lbs. of water, 3900 lbs. of sodium sulfate and, finally 30 lbs. of 93% sulfuric acid to bring the pH of the slurry down to 7.5-8.0. The mix tank agitator was kept running throughout the addition of these materials at such a rate that aeration of the slurry was minimized. From the mix tank the compounded slurry was pumped through the vacuum tank for deaeration at an absolute pressure of 50-75 mm. mercury. Finally the deaerated slurry was pumped through line 33 fitted with a steam jacket, which

preheated the slurry to 125° F. before it entered the nozzles of the spray drying tower.

The following procedure may be used for producing a detergent of the phosphated heavy duty type: A high active slurry containing 9263 lbs. water, 1396 lbs. sodium sulfate and 5764 lbs. sodium alkyl aryl sulfonate was heated to 100-110° F. and pumped to the mix tank. In this instance the weight ratio of sodium alkyl aryl sulfonate to sodium sulfate is 4.1 to 1. To meet product specification it was necessary to bring this ratio down to 3.4 to 1 by adding batchwise 300 lbs. of sodium sulfate. This slurry was run into another mix tank where it was mixed continuously with 8564 lbs. of commercial sodium tripolyphosphate and 4752 lbs. water. The temperature of the mix quickly rose to 115-125° F. due to the hydration of the phosphate. After being continuously pumped through the vacuum chamber the slurry was preheated to 185-190° F. just before entering the nozzles of the spray drying tower. A spray dried product resulted which had a density in excess of 0.30 gm. per cc.

I claim:

The method of producing a bead form detergent that includes dispersing an air containing aqueous slurry of the detergent into the atmosphere within a deaerating zone in finely divided form, maintaining said zone at a sub-atmospheric pressure below about 250 mm. Hg absolute to remove air from the slurry, said sub-atmospheric pressure tending to cool the slurry by evaporation of moisture therefrom, then passing the slurry, including all ingredients thereof which enter the deaerating zone except for air and vapors removed in said zone, into an independent spray drying zone and therein spray drying the deaerated slurry to produce the detergent in high density bead form, and heating said slurry in advance of its entry to the spray drying zone to compensate for the cooling effect of said sub-atmospheric pressure and thereby prevent thickening of the slurry by said cooling, the detergent slurry fed to the deaerating zone having a temperature between about 110° F. and 160° F. whereby maximum deaeration is effected, and the heating of the slurry in advance of its entry to the spray drying zone being sufficient to maintain a slurry temperature between about 120° F. and 190° F. immediately prior to entry of the slurry into the spray drying zone whereby a spray dried product of maximum density is obtained.

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