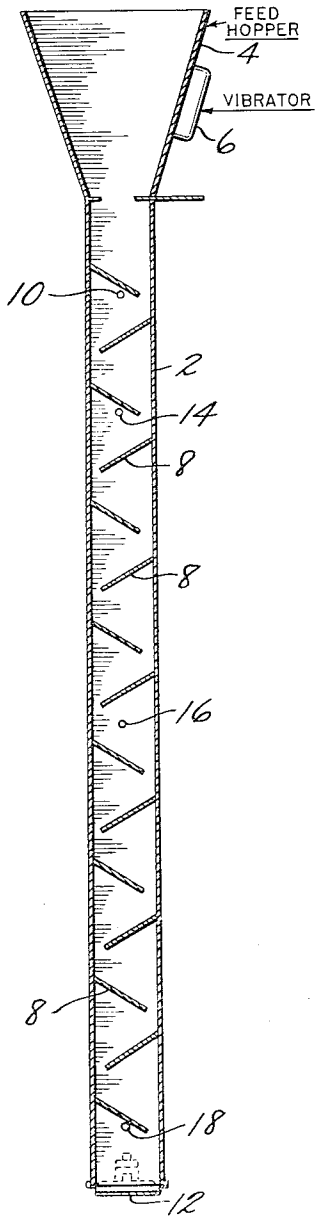
Aug. 9, 1955 F. G. PACKARD 2,715,110

METHOD FOR THE PRODUCTION OF A GRANULATED SOAP PRODUCT

Filed June 13, 1952 2 Sheets-Sheet 1

FIG. 1.



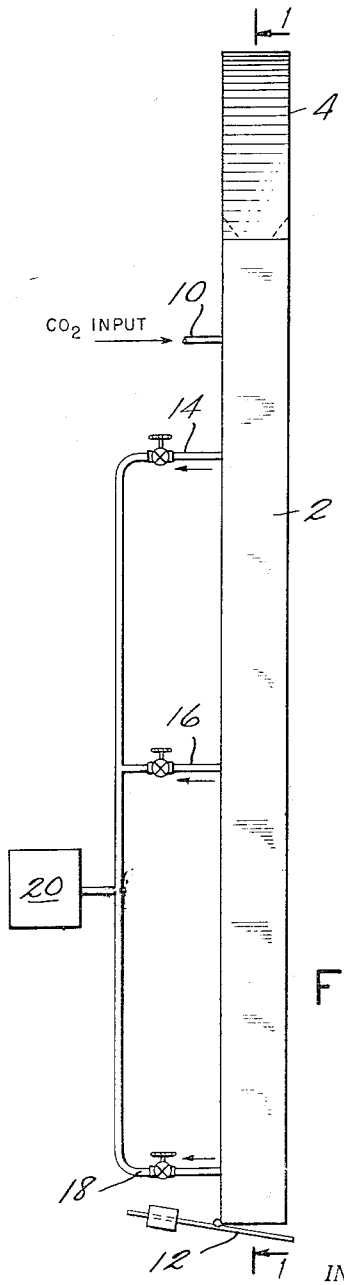


FIG. 2.

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METHOD FOR THE PRODUCTION OF A GRANULATED SOAP PRODUCT

Filed June 13, 1952 2 Sheets-Sheet 2

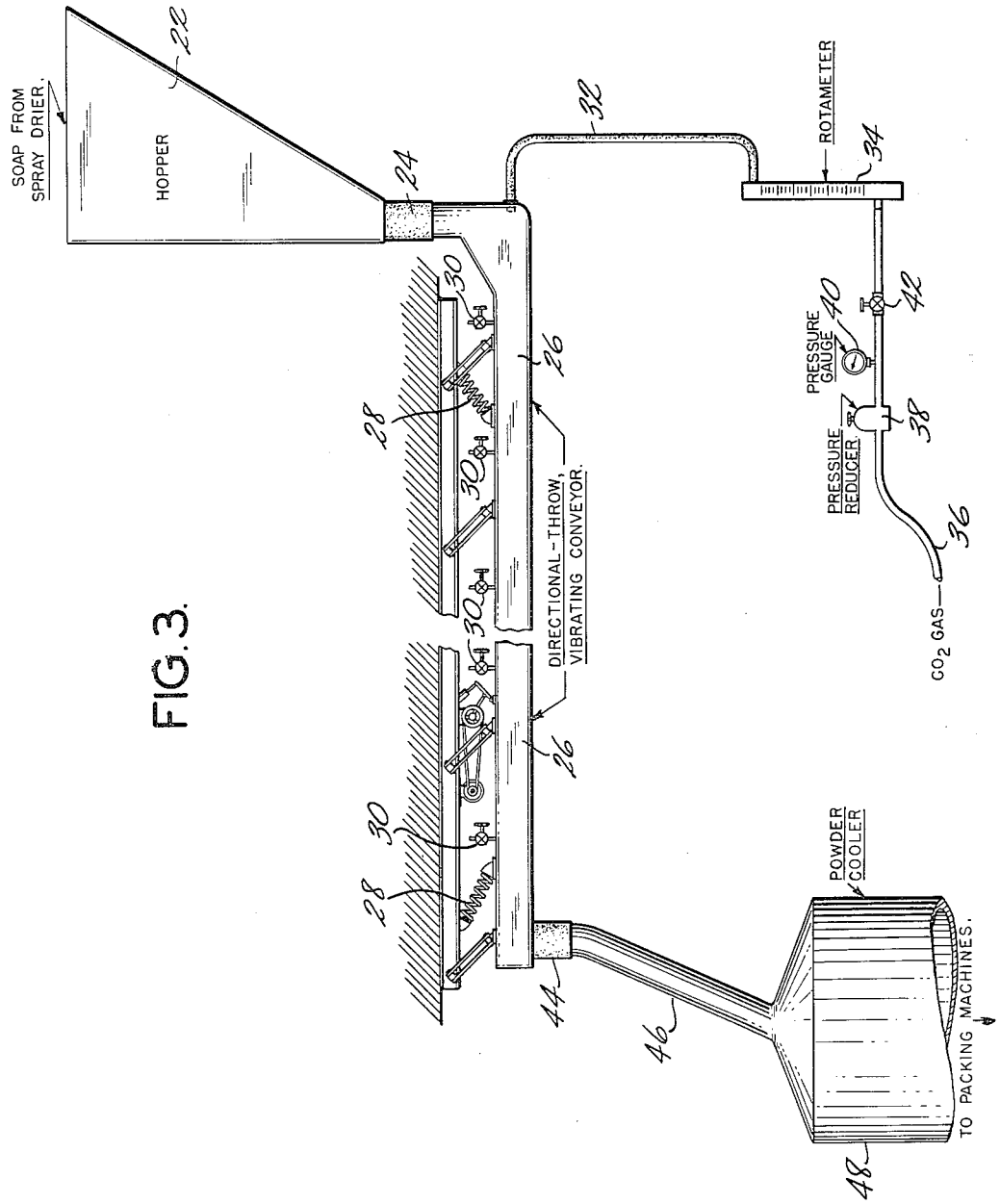


FIG. 3.

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2,715,110

METHOD FOR THE PRODUCTION OF A GRANULATED SOAP PRODUCT

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Application June 13, 1952, Serial No. 293,235

5 Claims. (Cl. 252—109)

This invention relates to a method for the production of a non-agglomerating granulated soap product.

Granulated soap products, such as spray-dried soap particles especially when freshly packed, exhibit a tendency to gel or agglomerate when poured into hot water. The aggregates of sticky soap thus formed are difficult to disperse and dissolve.

The tendency of spray-dried soaps to agglomerate has been demonstrated in the laboratory by pouring the soap on the surface of water, at 120° F., in a dishpan, agitating the water gently by hand for a short time and observing the agglomerates floating on the water. This method, although representing the condition that might prevail in actual usage, does not lend itself to the measurement of comparative degrees of agglomeration, and it is thus difficult to evaluate the characteristics of different soaps by such a test unless they are tested simultaneously. A modified test incorporating mechanical agitation and a controlled method of adding the soap power was, therefore, developed. In this test, the time required to disperse the soap agglomerates was taken as a measurement of the degree of agglomeration.

In this test, a dishpan was filled with nine quarts of water, heated to 120° F. An agitating paddle rotating at 45 R. P. M. was immersed in the water so that there was no vortex on the surface of the water. Thirty-five grams of the soap was poured through a funnel onto a deflecting plate and was thus spread over the surface of the water. The agitating paddle was run for 10 seconds after pouring the soap in the funnel, and it was then shut off. The appearance of the soap was then classified as heavy gel, moderate gel or loose floc. After waiting for 30 seconds, the agitating paddle was started again and the time required to disperse or dissolve the gel or floc was noted.

Since agglomeration is apparently caused by particles of soap melting or fusing together, the possibility of preventing agglomeration by intimately mixing a finely pulverized inorganic salt with a spray-dried soap was considered as a possible solution to the problem. The following materials were used for this purpose at a level of 5% of the total weight of soap powder, but no measurable improvement in agglomerating characteristics was obtained:

$NaCl$
Na_2SO_4
$Na_4P_2O_7$
Na_3PO_4
Na_2CO_3
$NaHCO_3$

Solutions of these materials were also sprayed onto the soap particles and the product subsequently was oven-dried to remove excess moisture. Little, if any, improvement in agglomerating properties resulted from this treatment, however.

It has now been found, in accordance with this invention, that the agglomerating tendencies of silicate-containing graular soap products can be eliminated by treating the substantially dry granules of soap with carbon dioxide gas. The treatment with carbon dioxide gas results in the precipitation of free silica particles on the surface of the granules, which it is found minimizes the agglomerating tendencies of the soap.

The presence of inorganic builders such as phosphates in the soap formulation is significant in obtaining a non-agglomerating product by carbonation, although the formation of free silica is not markedly affected when these materials are eliminated from the formula. Experimental soap formulations having varying amounts and proportions of inorganic builders including silicates, sodium carbonate and tetrasodium pyrophosphate all show a tendency to agglomerate when the freshly prepared powders are added to hot water at temperatures of from 120 to 150° F. After the same powders have been exposed to an atmosphere consisting essentially of carbon dioxide for approximately 10 seconds, agglomeration is, from a practical standpoint, eliminated.

Sufficient carbonation to effect the surface formation of free silica is necessary to insure practical non-agglomerating properties in current soaps. This requires the absorption of carbon dioxide by the finished powder. It has been found that amounts of free silica formed in accordance with the invention have no significant effect upon the use properties of the product.

It has been established that if there is no silicate present in the soap formulation, treatment with CO_2 gas does not improve the agglomerating characteristics of the soap. The amount of silicate which may be present in the soap varies from about 5.0% by weight to about 20.0% by weight. Any of the well-known silicates commonly used in soap formulations may be employed, for example, the sodium silicates.

It is also desirable to incorporate a phosphate in the soap formulation. The incorporation of the phosphate results in the desirable feature that the dispersing time of the soap agglomerates, if any, is greatly reduced.

Well-known phosphates commonly employed in soap formulations may be used, such as trisodium phosphate, tripolyphosphate and tetrasodium pyrophosphate.

The invention will be further illustrated by reference to the accompanying drawings, in which,

Figure 1 is a front view in elevation and in section, taken on line 1—1 of Figure 2, of a cascade tower for treating soap granules with CO_2;

Figure 2 is a side view in elevation of the cascade tower shown in Figure 1; and

Figure 3 shows another apparatus which may be used to treat soap granules with CO_2 in which the soap granules are passed through an atmosphere of CO_2 gas between the spray drier and the cooling tower.

Referring specifically to Figure 1, the tower **2** is provided with a feed hopper **4** for the soap granules, having a vibrator **6** attached thereto which assists the flow of the granules through the feed hopper. The tower contains a plurality of shelves or baffles **8**, which may be at an angle, and which serve to provide a cascading effect to the soap granules passing downwardly through the tower.

Carbon dioxide gas is introduced through the line **10**, preferably at a point below the first shelf, as shown in Figure 2, in order to reduce the leakage of CO_2 from the top of the tower. Since the CO_2 gas is heavier than air, it will pass downwardly through the tower. Gas losses from the bottom of the tower are minimized by counterweighted hinge closure **12**.

The rate of CO_2 gas flow into the tower may be measured by a calibrated rotameter in the line **10** (not shown). The gas concentration at several locations in the tower may be determined by withdrawing samples through the lines **14**, **16** and **18** into a CO_2 concentration measuring apparatus **20**, as shown in Figure 2. The measuring apparatus **20** compares the thermal conductivity of the

sample with that of air by means of a calibrated balanced bridge, as is well known in the art.

Figure 3 shows an alternative arrangement for treating the soap granules with CO_2 gas in which a hopper **22** is mounted below the conventional spray drier and receives the dried soap granules. The granules pass through a flexible rubber connection **24** into an enclosed directional-throw vibrating conveyor **26** which is supported by the springs **28** from the ceiling of the building.

A plurality of sampling ports **30** are provided on the top of the conveyor in order that the CO_2 gas concentration at various locations in the conveyor housing may be measured. A flexible hose **32** is connected to the right-hand end of the conveyor as viewed in Figure 3 and at its opposite end to a rotameter **34** which is used to control the volume of CO_2 gas passing into the vibrating conveyor. A CO_2 input line **36** is connected to the rotameter and to any convenient source of CO_2 gas and is provided with a pressure reducer **38**, a pressure gauge **40** and a valve **42**.

When the granules reach the left-hand end of the vibrating conveyor as viewed in Figure 3, they pass through a flexible connection **44** into a duct **46** connected to a powder cooler **48**. From the powder cooler **48**, the cooled granules are conveyed to packing machines, not shown.

The invention will be further illustrated by reference to the following specific examples:

EXAMPLE 1

A soap powder having the following formula:

	Percent
Fatty acids	55.00
Anhydrous soap	59.54
Tetrasodium pyrophosphate	7.00
Sodium carbonate	2.50
Sodium silicate	12.00
Sodium chloride	4.42185
Glycerin	0.31
Perfume	0.04
Moisture	14.00
Miscellaneous	0.02815

was introduced into the tower shown in Figures 1 and 2 at a rate of 2,550 pounds per hour. CO_2 gas was then introduced into the tower at the rate of 37 pounds per hour, which corresponds to 1.45% CO_2 calculated on the powder weight. The CO_2 concentration in the tower was 58% at the top, 27% in the middle, and 13% at the bottom. It was found that by treating the powder for from 7 to 9 seconds under these conditions, a substantially non-agglomerating powder was produced. The powder entered the tower at a temperature of 87° F. and flowed from the bottom thereof at 100° F. indicating an appreciable heat of reaction.

EXAMPLE 2

A powder having the same formula as that used in Example 1 was introduced into the tower shown in Figures 1 and 2 at a rate of 4,100 pounds per hour. CO_2 gas was introduced into the tower at the rate of 28.8 pounds per hour, corresponding to 0.7% CO_2 calculated on the powder weight. The CO_2 concentration in the atmosphere in the tower was 44% at the top, 16% in the middle and 2.5% at the bottom.

Operating under these conditions, a powder having much less tendency to agglomerate was produced.

EXAMPLE 3

A powder having the same formula as that used in Example 1 was introduced into the tower shown in Figures 1 and 2 at the rate of 1,275 pounds per hour. CO_2 gas was introduced into the tower at the rate of 13.7 pounds per hour, corresponding to 1.08% CO_2 calculated on the powder weight. The CO_2 concentration in the atmosphere of the tower was 42% at the top, 27% in the middle and 13% at the bottom.

Comparison of these results with those obtained in Example 2 above indicates that better stripping efficiency is obtained when the tower operates close to maximum capacity.

EXAMPLE 4

To ascertain the effect obtained when the tower was doubled in height, a powder having the same formula as that of Example 1 was passed through the tower shown in Figures 1 and 2 twice. The powder was introduced into the tower at the rate of 1,275 pounds per hour on the first pass and 1,280 pounds per hour on the second pass. CO_2 gas was introduced into the tower at the rate of 13.7 pounds per hour on the first pass and 2.0 pounds per hour on the second pass corresponding to 1.08% CO_2 on the powder weight of the first pass. The gas was added on the second pass to simulate conditions halfway down a 20 foot tower. The CO_2 concentration in the atmosphere in the tower was 42% at the top on the first pass and 11% on the second pass, 27% at the middle on the first pass and 5% on the second pass, and 13% at the bottom on the first pass and 1% on the second pass.

The powder was substantially non-agglomerating even though only 1.08% CO_2 was used. That improved stripping may be anticipated in a higher tower is shown by the lower CO_2 content of the air at the bottom of the tower on the second pass.

EXAMPLE 5

A series of formulations was prepared wherein sodium carbonate and tetrasodium pyrophosphate were eliminated from the product. The products were carbonated using 3% CO_2 and were subsequently tested for agglomeration and the presence of free SiO_2. The results indicate that the elimination of sodium carbonate and tetrasodium pyrophosphate does not markedly affect the formation of silica, but reduces the improvement in agglomerating characteristics that results from carbonation. The elimination of sodium carbonate alone has no adverse effect. Test results using various soap formulae are as follows:

Analytical (percent)

	Na_2CO_3	$NaHCO_3$	$Na_4P_2O_7$	Free SiO_2
Standard Soap (percent):				
Soap, 61.70				
Sodium Silicate, 11.70				
NaCl, 3.86				
Tetrasodium pyrophosphate, 7.23				
Na_2CO_3, 2.08				
H_2O, 14.00				
Untreated	1.72	0.92	7.36	0.12
3% CO_2 treated	7.13	1.18	7.25	5.21
Soap without Na_2CO_3:				
Soap, 63.20				
Sodium Silicate, 12.00				
NaCl, 3.95				
Tetrasodium pyrophosphate, 7.40				
Na_2CO_3, None				
H_2O, 14.00				
Untreated	0.48	0.15	7.18	1.20
3% CO_2 treated	4.22	0.53	6.74	4.63
Soap without tetrasodium pyrophosphate:				
Soap, 67.20				
Sodium Silicate, 12.78				
NaCl, 4.21				
Tetrasodium pyrophosphate, None				
Na_2CO_3, 2.26				
H_2O, 14.00				
Untreated	0.84	0.34		0.14
3% CO_2 treated	6.60	1.60		5.77
Soap without Na_2CO_3 or tetrasodium pyrophosphate:				
Soap, 68.20				
Sodium Silicate, 14.12				
NaCl, 4.26				
Tetrasodium pyrophosphate, None				
Na_2CO_3, None				
H_2O, 14.00				
Untreated	0.99	0.23		0.12
3% CO_2 treated	4.89	0.96		4.81

Agglomerating tests

	Appearance of Soap in Dishpan	Time to Disperse, secs.
Standard Soap:		
Untreated	Heavy gel	40
3% CO_2 treated	Loose floc	0-3
Soap without Na_2CO_3:		
Untreated	Heavy gel	40
3% CO_2 treated	Loose floc	3
Soap without tetrasodium pyrophosphate:		
Untreated	Heavy gel	40
3% CO_2 treated	Light gel	20
Soap without NA_2CO_3 or tetrasodium pyrophosphate:		
Untreated	Heavy gel	35
3% CO_2 treated	Moderate gel	20

EXAMPLE 6

A series of samples was prepared by exposing weighed amounts of fresh soap, having a formula the same as that employed in Example 1, to varying amounts of carbon dioxide in order to determine the minimum amount of carbonation required to obtain a non-agglomerating product. Carbon dioxide in the form of Dry Ice was added to the soap in the flask and was shaken until the carbon dioxide was completely vaporized. The agglomerating characteristics of the several samples were then determined by the dishpan test. This series of tests indicated that it was necessary for carbonation to take place to the extent that the product contained approximately 1.5% free silica. Large amounts of silica gave a non-agglomerating product, but excessive quantities might prove to be objectionable in other use tests. The chemical changes occurring in the standard soap formulation of Example 1 at different stages of carbonation are shown in the following table:

Amount of CO_2 Absorbed Based on Weight of Soap	Free Silica	Na_2CO_3	$NaHCO_3$	pH of 0.2% Solution
	Percent	*Percent*	*Percent*	
0 (Control)	Nil	2.82	Tr.	10.32
0.3%	0.2	3.59	Tr.	10.30
0.7%	1.3	3.36	0.88	10.22
1.0%	1.6	4.00	0.88	10.19
1.3%	2.0	4.43	1.11	10.17
1.6%	2.7	4.18	1.60	10.09
2.3%	3.7	3.83	2.98	10.03

It is apparently necessary to neutralize some of the excess sodium oxide in the silicate before any significant amount of SiO_2 is precipitated indicatng that higher ratio silicates would be desirable. Batches prepared using (1) $Na_2O:(2.71)SiO_2$, and (2) $Na_2O:(3.25)SiO_2$ substantiated this, non-agglomerating products being obtained with lesser amounts of CO_2 when the 3.25 ratio silicate was used.

The precipitation of free silica by carbon dioxide is probably a surface reaction. This was demonstrated by carbonating a standard soap product and then removing part of the outer surfaces of the particles by gently rubbing the particles on a fine mesh screen. The pH of the 0.2% solutions of the surface powder was less alkaline than that of the powder remaining on the screen as shown by the following figures:

	pH of 0.2% solution
Untreated powder	10.30
Surface powder removed	9.91
Residue, after partial removal of surface powder	10.05

Precipitation of free silica in the soap in the crutcher prior to soap drying was found to be less effective than exposing the finished powder to CO_2 since it results in the formation of a coarse silica that is rapidly deposited in the dishpan as the soap is dissolved.

EXAMPLE 7

To evaluate the effect of phosphates in the soap formulations of the invention, a series of six formulations were prepared using a soap having the same formula as that of Example 1 except that 7% of various phosphates were added to the formulations.

After similar treatment with CO_2 gas, the various soap formulations were analyzed and the dispersing time of the soap granules in accordance with the standard test hereinbefore set forth was measured. The results are as follows:

Formula	CO_2 level	Percent Free SiO_2	Percent Na_2CO_3	Percent $NaHCO_3$	Dispersing Time, sec.
1. Tetrasodium pyrophosphate	None (control)	Nil	2.82	Nil	35 (gel).
2. Tetrasodium pyrophosphate	2.3%	3.7	3.83	2.98	0 (floc).
3. Trisodium phosphate	None (control)	Nil	3.57	Nil	40 (gel).
4. Trisodium phosphate	2.0%	1.58	6.48	1.18	10 (floc).
5. Tripolyphosphate	None (control)	Nil	3.40	Nil	25 (gel).
6. Tripolyphosphate	2.0%	3.24	4.12	3.17	7 (floc).

From the above results, it is seen that when trisodium phosphate was used in place of tetrasodium pyrophosphate, the free silica formation during treatment with CO_2 gas was retarded to a considerable extent, although the product was non-agglomerating. These data indicate that the alkalinity of the product during gaseous treatment has some relation to the reaction, and it is believed that the stability of the sodium silicate present in a soap formulation is related to the alkali content in the product in which it is used. It is to be expected that a very alkaline phosphate would have a greater affinity for CO_2 and thus the formation of free silica would be retarded.

The liberation of free SiO_2 by CO_2 gas treatment is effective with greater ease by increasing the $Na_2O.SiO_2$ ratio in soap to higher values of SiO_2 relative to the Na_2O and it has been found that the free silica development is proportional to the formation of $NaHCO_3$. Using the soap formulation of Example 1, it was found that the free silica development began when the $Na_2O.2SiO_2$ ratio of the sodium silicate present in that soap formulation reached approximately 3.16.

EXAMPLE 8

Two experimental soap formulae were prepared. The finished formulae were as follows:

	A	B
	Percent	*Percent*
Fatty Acids	55.00	55.00
Anhydrous Soap	59.54	59.54
Tetrasodium Pyrophosphate	7.00	7.00
Sodium Carbonate	3.50	4.50
Sodium Silicate	13.00	17.75185
Sodium Chloride	6.42185	0.67
Glycerin	0.31	0.31
Perfume	0.04	0.04
Moisture	10.00	10.00
Misc	0.02815	0.02815

CO_2 gas treatment of these products was completed in the apparatus shown in Figure 3 on a full production scale. Production rates ranged in the vicinity of 10,000 pounds per hour and CO_2 was introduced into the conveyor at rates varying from 140 pounds per hour to 250 pounds per hour. The product obtained at the lowest CO_2 feed rate was not satisfactory.

Operating at the rate of 200 lbs. of CO_2 per hour with the soap of formula B being conveyed at the rate of 10,000 lbs. per hour produced a product having approximately 2.25% SiO_2 on the surface of the soap particles. Operating at the higher rate of 240 lbs. of CO_2 per hour with the soap of formula A produced a product having 1.22% SiO_2 on the surface thereof.

Results of several production runs are as follows:

Test No.	Formula	CO_2 feed rate, lbs./hr.	Percent free SiO_2 developed	Dispersing Time (agglomerating test)
1	A	None (control)	0.10	35 sec. (gel).
2	A	240	1.22	5 sec. (floc).
3	B	None (control)	Nil	46 sec. (gel).
4	B	140	1.23	35 sec. (gel).
5	B	200	2.25	5-10 sec. (floc).

It has been indicated heretofore that the extent of the carbon dioxide treatment should be such as to give a product having satisfactory non-agglomerating properties. This will depend to a large extent upon the amount of the silica on the surface of the product and also upon the depth of the silica formation. In general, products having less than about 1% silica deposited primarily on the surface thereof do not show satisfactory non-agglomerating properties from a commercial acceptance point of view.

The length of treatment, the concentration of carbon dioxide in the treating atmosphere and the amount of carbon dioxide in relation to the soap product being treated are inter-related and are not critical as long as the requisite amount of silica is obtained. High carbon dioxide concentrations permit treating times as short as 3 seconds, and there is no critical upper limit to the treating time. The concentration of carbon dioxide, when adjusted to the treating time, may vary from 40% to 100% in the treating atmosphere.

In the above disclosure certain soap formulations have been described, but it will be understood that the process can be applied to any silicate containing soap. In the above example the soap consisted essentially of the sodium salt of fatty acids of tallow, but the potassium salt may be employed, and the fatty acids may be derived from other animal and vegetable fats as is well known in the soap industry.

In addition to containing a silicate within the range mentioned heretofore, the soap may be "built" with any of the common builders such as the phosphates mentioned heretofore, carbonates and in general alkaline salts, and other ingredients. The proportions of soap to silicate and the proportions of soap to builders is not critical and the invention is applicable to any of the formulations in which soap is the primary ingredient and contains a silicate.

The silicate is usually the sodium silicate but any other alkali metal may be used, having the formula, using sodium as illustrative, $(Na_2O)_x.(SiO_2)_y$ where x and y may be in the range of about 1:1 to 1:3.25 or higher.

It is also well known that freshly spray-dried soap, although dry in appearance, contains about 5 to 20% moisture and such soap products can be treated in the process.

It will be obvious that many soap formulations may be treated in accordance with the invention and a wide variety of equipment and processing techniques will be obvious to those skilled in the art. These are intended to be included within the scope of the invention when they or their equivalents are embraced within the following claims.

I claim:

1. The method for making a granular soap product rapidly dispersible in water and substantially free from gel forming and agglomerating characteristics comprising preparing an alkali-metal silicate-containing soap and treating said soap material when in granular form with carbon dioxide in a concentration and for a time sufficient to form silica on the surfaces of said granulated soap particles but insufficient to make the particles substantially water-insoluble.

2. The method for making a granular soap product rapidly dispersible in water and substantially free from gel forming and agglomerating characteristics comprising preparing an alkali-metal silicate-containing soap, spray drying said soap material and treating said soap material when in a spray-dried granular form with carbon dioxide for a time sufficient to form silica on the surfaces of said granulated soap particles but insufficient to make the particles substantially water-insoluble.

3. The method for making a granular soap product rapidly dispersible in water and substantially free from gel forming and agglomerating characteristics comprising preparing an alkali-metal silicate-containing soap, treating said soap material when in granular form with carbon dioxide for a time sufficient to form silica on the surfaces of said granulated soap particles but insufficient to make the particles substantially water-insoluble and packaging said treated granular soap product.

4. The method for making a granular soap product rapidly dispersible in water and substantially free from gel forming and agglomerating characteristics comprising preparing a soap containing an alkali-metal silicate and treating said soap material when in granular form with carbon dioxide for a time sufficient to form at least about one per cent of silica primarily on the surface of said soap particles but insufficient to make the particles substantially water-insoluble.

5. A method for making a substantially dry, granular, alkali-metal silicate-containing soap product rapidly dispersible in hot as well as cold water and substantially free from gel-forming and agglomerating characteristics which comprises treating the substantially dry soap granules with carbon dioxide in a concentration and for a time sufficient to form silica on the surfaces of the granulated soap particles but insufficient to make the particles substantially water-insoluble.

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