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SOLID SARCOSINATE PARTICLES AND METHOD OF PREPARATION THEREOF

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This invention relates to new improved particles of salts of N-acylated sarcosine (hereafter called sarcosinates, fatty acid sarcosinates, or salts of fatty acid sarcosinates) in the solid state and to a method of producing such particles. More particularly it relates to hollow substantially dust-free, dry fatty acid sarcosinate beads which are, largely because of their physical shape and uniformity of size, more readily soluble in aqueous media than sarcosinate particles made by conventional drying methods, and to a process for producing such beads by atomizing into a drying gas under certain conditions, a solution, dispersion, or slurry of said sarcosinate.

Dental preparations, such as toothpastes and powders, containing sodium N-lauroyl sarcosinate and/or certain homologues thereof as the active ingredient possesses the property of inhibiting acid formation in the mouth for extended periods of time after brushing the teeth therewith.

It has been found desirable in some instances to prepare the active ingredient in solid particulate form for incorporation in dental preparations. In solid form the sarcosinates are more stable and they are more suitable for incorporation in tooth powders than in the form of an aqueous solution. Also, when the product must be transported shipping costs are lower. Because, in the usual manufacturing process the sarcosinate is obtained in solution, a drying step is necessitated. Due to the peculiar properties of sarcosinates the drying of solutions thereof presents a troublesome problem.

The sarcosinates are, in general, low melting compounds (the melting point of sodium N-lauroyl sarcosinate for example is 293–302° F.) and consequently are easily fused during normal drying operations, making their obtention in particulate form a difficult task, since, when heated they tend to coalesce, especially on drying zone boundary surfaces. In addition aqueous solutions of some sarcosinates gel, e. g., sodium N-lauroyl sarcosinate gels at concentrations over about 40 percent by weight in water, at 70° F., and at concentrations over about 45 percent at 180° F., and sodium N-palmitoyl sarcosinate gels at concentrations greater than 36 percent at 70° F., and over 38 percent at 180° F. Such gels tend to block pipe lines and spray nozzles before atomization and will tend to adhere to drying zone boundary surfaces if present in the drying zone, thereby hindering production because a mass of sarcosinate gel is slow to dry. In addition, such a gel very strongly holds a few percent of water, and so sarcosinate obtained therefrom is often not really dry.

The sarcosinates are decomposed to fatty acids and sarcosine when heated in the presence of water to too high a temperature or when solutions are allowed to stand too long, especially at elevated temperatures.

Because of the sensitive nature of the sarcosinates, their low melting points, and gel-forming properties, the ordinary commercial methods of drying materials have been unsatisfactory when applied to them. Such methods, which shall be referred to as conventional drying

processes, includes roll or drum drying, tray drying, countercurrent spray drying and even cocurrent spray drying. Crystallization, followed by air drying of the crystals, is uneconomic. Applicant's process on the other hand enables one to secure by a commercially feasible method, an attractive, useful, dry, readily soluble, solid particulate sarcosinate containing a minimum of fatty acid material (fatty acids and fatty acid soaps).

In accordance with the present invention a solution, dispersion or suspension, consisting essentially of a fatty acid sarcosinate, the acyl radical of which contains from 10 to 18 carbon atoms, in water, is atomized into a heated drying gas at the top of a vertically elongated zone, while cooling air is admitted into the zone at a point below that of atomization, to rigidify the dried sarcosinate particles. The dried fatty acid sarcosinate particles are withdrawn at the bottom of the zone as is a mixture of spent drying gas and cooling air.

The sarcosinates contemplated as within the invention, are the salts of amides formed from sarcosine and either straight chain saturated carboxylic acids or those fatty acids containing 1 or 2 double bonds, all of which acids are within the 10 to 18 carbon atom range, e. g., lauric acid, myristic acid, palmitic acid, oleic acid, coconut oil fatty acid and split hydrogented tallow fatty acid, especially those which, either because of their relatively low stability, low melting points or tendency to gel, or combination of such characteristics, cannot be dried in the conventional ways, into discrete, non-dusting particles.

The cationic substances that are combined with the sarcosine amide to form salts may be selected from the alkali metals or nitrogen containing radicals. More particularly they may be sodium, potassium, ammonium, amine, mono-, di- or tri-ethanolamine salts. However it is preferred that the sarcosinates to be subjected to the invented processes should be the sodium salts of sarcosinates whose fatty acyl groups are saturated and contain from 12 to 16 carbon atoms.

The sarcosinates may be made by condensing a fatty acid chloride or a mixture of fatty acid chlorides with sarcosine in the presence of a hydrogen halide acceptor, e. g., caustic soda, soda ash, magnesium hydroxide, trimethylamine, separating out the resulting amide, acid washing and subsequently neutralizing the acid amide with a base containing the desired cation. When intended for use in dental preparations for its acid inhibiting effect the sarcosinate should be nearly free of fatty acid materials (fatty acid soap and fatty acids) because of the deactivating properties thereof. Ordinarily the sarcosinate is obtained in solution and to promote economical drying such a solution (but it may be a dispersion) should usually be of as high a sarcosinate content as possible. In this specification and in the claims the word solution as applied to sarcosinates to be spray dried includes true solutions, colloidal suspensions, suspensions, dispersions and slurries, and is exclusive of gels. Since at higher concentrations, e. g., above 40% sarcosinate in water at room temperature, in the case of sodium n-lauroyl sarcosinate (also called sodium N-lauroyl sarcosine), a gel is formed, such a gelation concentration is the upper feasible solution concentration limit of the invented process since above that concentration the gel formed will block pipe lines and/or atomizing means. The addition of solvents, e. g., isopropanol, ethanol etc., is contemplated since they will often inhibit gel formation and enable one to utilize solutions of higher sarcosinate content, but manufacturing losses of such relatively costly solvents often make their use uneconomical. In addition it must be kept in mind that many solvents are flammable and hence their use may be hazardous. Some are poisonous and residues thereof in an incompletely dried product, intended

for dental use, would be dangerous. The addition or inclusion of inorganic salts, e. g., sodium sulfate, is contemplated but often it is desirable or necessary that the sarcosinate be obtained in an unadulterated, relatively pure state, and hence in those cases the inclusion of fillers will not be tolerated.

The relatively pure sarcosinate solution is heated or cooled to the proper atomization temperature, the selection of such temperature being determined primarily by the viscosity of the solution and its decomposition or dissociation rate thereat, and the holding time before atomization. In the case of sodium N-lauroyl sarcosinate it has been found that a 40% solution can be kept at 140° F. for one month without even 1 percent decomposition. Therefore solution temperatures below this amount are safe for that sarcosinate. Ordinarily the solution atomization temperature is almost as high as possible within the limits imposed by the factors already mentioned, inasmuch as by raising the initial solution temperature the dried material throughput can be increased slightly, less heat being required from the drying medium proper, and also because at higher temperatures the solution viscosity is lower, and consequently better spray rates and patterns are obtainable.

Because at the present time most dental preparation formulas containing sarcosinates require it to be relatively pure this specification will describe the drying of a sarcosinate solution which contains inorganic salts in only negligible amounts, if at all. However it should be understood that the process is just as applicable to the drying of solutions or dispersions of sarcosinates containing larger amounts of inorganic materials, and the drying of such solutions or dispersions is within the inventive concept and claims, especially so if they cannot be readily dried by the prior art methods hitherto employed for the preparation of dried fatty acid sarcosinates.

While it is evident that drying conditions must be set specifically for the particular compound being processed, because of different solubilities, stabilities (ammonium and amino compounds, for example, being more susceptible to decomposition), gel ranges, viscosities, product hardnesses and aggregative tendencies, generally the solution temperature immediately prior to atomization should be between 30° F. and 140° F., preferably 70° F. to 140° F., the solution, consisting essentially of sarcosinate and water, being at a concentration between 10 and 40 percent by weight of sarcosinate at which concentration there is no interfering gelation and the solution is not too viscous to be pumped and atomized or sprayed. To increase drying zone throughput usually solutions of concentrations between 30 and 40 percent will be employed where obtainable. The optimum concentration for a sodium N-lauroyl sarcosinate solution before drying is between 32 and 37 percent by weight, since at concentrations above 37 percent the viscosity of such a solution increases markedly.

The word solution, as previously indicated, is generally used to describe a true solution or suspension in water of a sarcosinate, but non-interfering solvents such as lower molecular weight alcohols, e. g., ethanol, isopropanol, may be present to increase sarcosinate solubility and/or decrease viscosity. The sarcosinate solution may contain adjuvant materials or fillers, additives or impurities which do not significantly detract from the utility of the product or seriously interfere with the invented process.

After heating, which may be done in a jacketed crutcher or other suitable heating device, or after cooling, if either heating or cooling is necessary, the sarcosinate solution is pumped to the atomizing device, which may be a conventional spinning disc, pressure nozzle, or other atomizing means.

The atomization of the solution is carried out at the top or upper portion of a zone of gaseous drying medium, the temperature of which medium at a point just above that of atomization, is between 250° F. and 600° F., usually between 375° F. and 500° F., and preferably between 400° F. and 475° F. Above the broad range of temperatures shown there is danger that fine droplets of solution will dry extremely rapidly and then be heated to a temperature at which they will decompose, discolor or char certain sarcosinates. Below that temperature range, low, uneconomic drying rates are usually encountered, and unless the time spent by a particle in the drying zone (holdup time) is increased, wet particles may be obtained.

The atomized sarcosinate solution falls through the drying zone, travelling in the same direction as does the drying gas, the rate of descent of the droplets being dependent on the force of gravity, velocity of the drying gas stream, particle size and specific gravity. As the droplets fall they dry, and the temperature of the drying gas decreases due to the transfer of heat from it to the droplets.

Almost as soon as the sarcosinate solution enters the drying zone in droplet form the droplet surface loses moisture and begins to harden, making coalescence with other droplets, with which it may come in contact, unlikely so long as the droplet surfaces are relatively hard.

The surface temperature of the particles being dried does not exceed the boiling point of water at the pressure of the drying zone as long as the particle contains some moisture, because, in the usual application of the invented process, when the temperature of the drying gas is within the temperature range specified, the mass transfer rate (of water to the surface of the drying particle) is greater than the heat transfer rate (of heat to the particle from the drying gas). Hence there is no danger of charring (the sarcosinates do not char at 212° F., and usually the drying zone is at approximately atmospheric pressure) while the particle is still evaporating moisture, or, to say it differently, while the mass transfer compensates for heat transfer. Nevertheless when the particle is dry its temperature can rise above 212° F. and can approach the temperature of the drying gas. If the gas was to be directed from the bottom of the drying zone to the top, the falling dried or surface-dried sarcosinate beads would encounter the hottest gas and so the chances of decomposition of the sarcosinate would be much greater. It is very difficult to control such a process and by such control prevent product degradation, and formation of fatty acid, while still drying material at a sufficiently rapid rate. On the other hand, since in the invented process the gas travels in the same direction through the drying zone as do the atomized particles (top to bottom), as the particles dry the gas temperature drops, and consequently the danger of product decomposition is considerably lessened and process control is facilitated.

In some instances, even when the drying air and sarcosinate particles pass through the drying zone in the same direction, the dry sarcosinate beads, because of their low melting points, may be at a temperature near or above that of fusion. In such a situation it is obvious that when those particles contact each other they will tend to stick together, forming aggregates. The boundaries or walls of drying zones, if no special steps are taken to cool them, will assume the temperature of drying gas in contact therewith. Consequently when dried solid sarcosinate particles contact such hot walls, if the wall temperatures are above that at which the sarcosinate melts, the product will fuse, losing its uniform, conveniently-sized discrete character and forming lumps or slabs which require grinding or pulverizing before being usable. Of course, the temperature of the drying gas can be lowered and this danger minimized, but in that case production rates must be cut.

It has been found that fusing of spray-dried particles into large aggregations can be minimized, if, after the

particles are dry, a blast of relatively cool air is directed onto said particles and/or onto the walls of the drying zone with which the dried particles might make contact. This should preferably be done in the lower part of the drying zone, after the particles have been dried, so that the addition of cool air will not lower the temperature of unspent drying gas and so adversely affect drying rates. The cool air, even if not directed on the zone walls, will mix with the hot spent drying gas and lower its temperature. This mixture will in turn be the medium contacting the lower zone bounding members and consequently zone wall temperatures will be decreased. The danger of particles melting thereon will be lessened because they will be cool, and consequently rigid, and will be contacting a relatively cool wall. To be effective the cooling air directed on the dried particles should be of a temperature lower than 100° F. and preferably lower than 50° F. While the use of cooling air of any temperature below that of the drying gas at the point of admission of cooling air will be helpful to some extent, the invention preferably contemplates the use of sufficient cooling air at a temperature low enough to rigidify the particles on which it is directed, i. e., at a temperature low enough to decrease the particle temperature to one at least below the sarcosinate melting point and preferably to a temperature at least below the lowest temperature at which the sarcosinate particles are tacky. It has been found that to avoid tackiness sarcosinate particles, e. g., sodium N-lauroyl sarcosinate particles, e. g., sodium N-lauroyl sarcosinate particles containing about 1% moisture, should be at about 200° F. or below when withdrawn. Usually, to secure a sarcosinate product at such a temperature it will be necessary to have the exit gas mixture at 190° F. or less. Obviously the higher the moisture content of the sarcosinate the lower must be its temperature at withdrawal and therefore the desirable exit air temperature is lower too but it will never be less than room temperature.

The humidity of the cooling air is not critical, but low-humidity air will increase slightly the rate of drying of the sarcosinate particles, and will not promote aggregation of hydroscopic materials, and therefore its use is advantageous.

The melting onto zone boundaries of the sarcosinate, while being dried, may be prevented by cooling the zone walls and product as described above and/or by "insulating" the drying beads from the walls with a circulating stream of relatively cool air.

Should hot particles of sarcosinate contact the zone boundary and, because of their plastic or molten state, stick there, eventually a large slab of sarcosinate would coat the zone wall. This would necessitate cleanouts because such a coating would decompose or char, and off-grade particles falling into correctly dried material would contaminate it. The avoidance of such contamination is especially important in the case of sarcosinate intended for use in dental preparations, where taste, color and purity are important factors.

To prevent buildup of the sarcosinate on the zone boundaries cooling air should be admitted tangentially into the drying zone preferably directed so as to pursue a path about the suspension of droplets and particles in the drying gas. Such cooling air performs two functions. It lowers the temperature of the droplets and particles nearest the zone boundary and so promotes solidification or hardening thereof and consequently minimizes the opportunity for such particles to stick to the said boundary. Because of its tangential admission the cooling air pursues a path close to the drying zone boundaries; it does not mix excessively with the descending drying gas and so does not seriously adversely affect the gas temperature and sarcosinate drying rate. The vertically elongated zone should be of circular or elliptical cross-section or of some other such shape which will minimize the degree of mixture of drying air and circumferential cooling air. The cooling gas also serves to prevent the particles, whether melted or solid, from contacting with the zone walls. Incidentally, such cooling air, near the point of removal of gases from the drying zone, will mix with the drying gas and other admitted cooling air intended for direct cooling of dried particles, and will lower the temperature of such mixture, thereby aiding in rigidifying the sarcosinate particles.

While the tangentially admitted air may be as warm as the drying gas and still perform its "insulating" function usefully, to secure the other advantage mentioned above the air temperature will usually be between 35° F. and 100° F. Ordinarily air at room temperature will be employed.

The mixture of spent drying gas and cooling air withdrawn from the drying zone will normally be from 100° F. to 250° F. and preferably 150° F. to assure that the sarcosinate particles are rigidified when they too are withdrawn. The word "spent" in this specification denotes drying gas whose drying power is less when it leaves the drying zone than it was when it entered, because of moisture vapor pickup by the gas, and a lowering of its temperature. Gas unsaturated with moisture vapor is, of course, included within the meaning given.

The term "rigidified" describes particles that are sufficiently firm so that they will not adhere one to the other when in light contact and whose interiors are not hot enough to cause warming and subsequent softening of the particle surfaces after withdrawal from the drying zone, under normal handling conditions. It is preferred to have the whole particle at a temperature below that at which it becomes tacky, usually at a temperature below its fusion point, but it is recognized that satisfactorily rigidified particles can be made which are warmer internally than the sarcosinate at its softening or fusion points, providing that the particle surface is rigid and emits heat as rapidly as it is conducted thereto from the particle interior.

The sarcosinate beads made as described above are discrete particles of generally uniform size usually containing at least 80 percent of sarcosinate, 12 percent or less water and less than 8 percent of fatty acid materials, a large percentage of which particles is in hollow substantially spherical shape. The spheres often contain one or more holes in an otherwise continuous surface. While the sizes of the beads can be adjusted by varying the spraying pressure or spinning disc speed, crutcher mix viscosity, feed rate, solids content and other conditions, to enable one to secure substantially all the spray dried particles in particular lower particle size ranges, e. g., over 90 percent by weight in the 10 to 150 micron diameter range, it is within the scope of this invention to utilize its processes for the manufacture of products of larger size particles too, for instance those containing particles mostly within the 100 to 500 micron range. However for tooth powder formulation the range will be from 10 to 150 microns diameter, and, where the avoidance of dust is highly important, 20 to 150 microns. Particle sizes may be adjusted for various reasons, e. g., to minimize dust, obviate the necessity of grinding, make attractive products, promote rapid solution, etc. Thus, particles of 10 to 150 microns, preferably 20 to 150 microns diameter, made by the atomizing-drying-cooling process described above may be incorporated in tooth powders by blending, without the necessity of grinding larger pieces of sarcosinate and without the excessive dusting that almost invariably accompanies such grinding or pulverizing. Tooth powders so made are relatively dust-free, of sufficiently fine appearance, and non-sifting.

If a sarcosinate solution was dried by one of the usual drying processes it would be necessary to grind or pulverize the resulting solid. If ground or pulverized to a size suitable for use in tooth powder it would be almost impossible to avoid having present in the powder a considerable amount of dust, more than in the case of mate-

rial dried by the invented process. Excessive dusting of a tooth powder containing sarcosinates results in irritation of the mucous membranes of the user and causes significant decreases in consumer acceptance. It also is objectionable to workers engaged in formulating or packing such material.

In cases where the dried sarcosinate is to be incorporated in tooth pastes it is necessary to dissolve it in water. It has been found that the products of this invention dissolve more rapidly than do size-reduced sarcosinates made by the previously mentioned conventional drying methods because it is necessary, to avoid dusting, to make the ground particles of larger sizes, since they are not as uniform in size as the particles of the invented process, and hence contain more finer (dusting) material. If the conventionally dried sarcosinates are pulverized they will contain many exceedingly fine particles. These will cause dusting and will, because of their small particle size, also agglomerate more readily than the invented products when placed in water; hence pulverized sarcosinates are more difficultly soluble than those made by the subject process. The particles made according to the described invention are generally of a more porous nature than those made by crystallization, drum drying or tray evaporation methods. This porosity, together with a hollow perforated bead construction, facilitates more ready solution than is attainable with sarcosinates dried by the other named processes.

The sarcosinate beads of this invention, being round, present relatively small bead to bead contact areas and therefore such particles tend less to aggregate and lump, even when warm or moist, than do sarcosinate particles of similar composition and of flatter non-spherical shapes, produced by grinding or pulverizing. The invented beads being non-aggregative, flow more readily and hence do not block filling machinery or feed lines, or retard filling rates as much as the size-reduced sarcosinates.

The fatty acid material content of sarcosinate beads intended for use in dental preparations must be kept low because, as has been previously stated, high fatty acid material content decreases the acid inhibiting activity of the sarcosinate and allows more mouth acid formation. It has been discovered that the soap content affects the particle solubility characteristics too, so that in the case of sodium N-lauroyl sarcosinate beads, those containing one part fatty acid material per 9 parts sarcosinate were noticeably slower to dissolve completely in water than beads consisting of the sarcosinate alone.

Contrary to expectations it has also been discovered that as the soap content of a sarcosinate solution increases the ease of drying by the invented process decreases. Thus, when a solution of sodium N-lauroyl sarcosinate containing one part by weight of fatty acid material (sodium soap, in this case) per 9 parts sarcosinate, was dried by the process of this invention the degree of drying secured was significantly less than that resulting when a solution containing less than one part soap per 99 parts sarcosinate was dried. Consequently, it is seen that to secure the same moisture content in finished products one must reduce the throughput of soap-containing material.

While the maximum fatty acid material concentration of the beads is dependent on the sarcosinate used, from the acid inhibiting activity, solubility characteristics and drying rate standpoints it should not exceed 10 percent by weight of the sarcosinate itself. Preferably it should be less than five percent, and ideally zero percent.

The following examples are given to further indicate the nature of the invention. It will be apparent that the examples are merely illustrative and the invention is not to be limited thereto.

All parts are by weight unless otherwise indicated.

Example I

A water solution at 60° F., containing 36 percent by weight of sodium N-lauroyl sarcosinate and less than 0.4 percent soap is atomized at the top of a drying zone into 475° F. drying air at the rate of 570 parts solution per hour. Both the drying air (air plus oil combustion products) and solution droplets or particles flow from top to bottom through the drying zone. The drying gas is sent through said zone at the rate of about 270 parts/minute. Zone holdup time (considering particles and drying air flow rates to be the same) is approximately 60 seconds. Cooling air at 60° F. is admitted to the drying zone tangentially at the rate of 170 parts/minute. Other cooling air at the same temperature is directed on the dried particles at the zone bottom at the rate of 170 parts/minute. The mixed gases are withdrawn from the drying zone at 190° F. and the dried product is removed through openings in the zone bottom after which it is conducted via pneumatic conveyor to a device for separating solid particles from carrier gas, e. g., a cyclone separator. The dried product from the separator is obtained at 200° F. The product rate is 195 parts/hour, inclusive of some sarcosinate adhering to walls at a portion of the tower where circumferential cooling air is not present. At portions of the tower where circumferential cooling air is admitted wall buildup is negligible.

The product obtained is examined microscopically and is found to be of the following size distribution.

Size Range, Diameters (Microns)	Weight Percentage of Particles Within Range
Below 10	0.01
10–20	0.4
20–40	1.6
40–50	1.6
50–60	1.0
60–80	1.7
80–160	16.2
160–320	44.0
320–500	33.5

Weight distribution of particles according to size was calculated in this and the following examples by multiplying the number of particles in each particular size range by the cube of the mean diameter of that range, totalling the said products, and dividing individual products by the sum of all products.

The product is of attractive appearance, light color, satisfactory taste and flows easily. The bead moisture content is 0.68 percent and soap content is approximately 1 percent. Its acid inhibiting activity determined by in vitro testing, is excellent, as great as that obtained from recrystallized sodium N-lauroyl sarcosinate. The discrete beads obtained are not tacky and do not lump. When ordinary countercurrent spray drying techniques are applied to the solution more than 20 percent by weight of the sarcosinate adheres to the tower walls and the collectable product made is hot and aggregates into lumps. When ground such sarcosinate is dusty and does not flow as readily as the dried atomized material described above. Since it forms lumps when added to the solvent, it is not as readily soluble in water at room temperature, in which it is dissolved to form a toothpaste intermediate, as is the invented product.

As the microscopic determination of particle size indicates, even the product made by the invented method is of too great a particle size for direct incorporation in quality toothpowders. However variation of the atomization conditions, increase in disc speed or nozzle pressure, decrease in crutcher mix solids content and viscosity, etc., decrease the particle size, other conditions remaining the same; more even distribution of circumferential cooling air decreases aggregation due to wall buildup and a product acceptable for toothpowder formulation is obtained.

Example II

A water solution at 60° F., containing 36 percent by weight of sodium N-lauroyl sarcosinate and less than 0.4

percent soap is atomized at the top of a drying zone into 450° F. drying air at the rate of 35 parts solution per minute. The drying air and solution droplets flow from top to bottom through the drying zone, the drying gas rate through the drying zone being about 4000 parts per minute. Zone holdup time is approximately 9 seconds. Cooling air at 60° F. is admitted to the drying zone tangentially at the rate of about 6000 parts per minute and other cooling air at the same temperature is directed at the same rate onto the dried particles at the zone bottom. The mixed gases are withdrawn from the drying zone at 150° F. and the dried product is removed at 160° F. The product obtained is of satisfactory color, taste and appearance and, as shown by the particle size tabulation below, may be incorporated in quality toothpowders without the necessity of grinding or pulverizing.

Particle Size Range, Diameters (microns)	Weight Percentage of Particles Within Range
Below 10	0.45
10–20	6.4
20–40	17.0
40–50	12.5
50–60	6.4
60–80	27.0
80–160	30.2
160–320	Nil
320–500	Nil

Example III

A water solution at 60° F. containing 15 percent of sodium N-palmitoyl sarcosinate and less than 0.2 percent soap is atomized at the top of a drying zone into drying air at the rate of 60 parts solution per minute. All process conditions, including air and product temperatures and rates are the same as in Example II. The product made is of good color, taste and appearance and may be incorporated in quality toothpowders without grinding or pulverizing.

It is of the following particle size distribution:

Particle Size Range, Diameters (microns)	Weight Percentage of Particles Within Range
Below 10	2.6
10–20	12.0
20–40	38.2
40–50	13.6
50–60	5.4
60–100	25.5
Over 100	Nil

The above invention has been described in conjunction with various illustrative examples thereof. It will be obvious to those skilled in the art that other variations and modifications of the invention can be made and various equivalents substituted therein without departing from the principles disclosed or going outside the scope of the specification or the purview of the claims.

Where, in the specification and claims the term "consisting essentially of" is used, as in the definition of the ingredients present in the compositions subjected to the processes of this invention or in the definition of the ingredients present in the compositions claimed, it is intended to exclude the presence of other materials in such amounts as to interfere substantially with the processes or to interfere substantially with the properties and characteristics possessed by the composition set forth, but to permit the presence of other materials in such amounts as will not substantially adversely affect the said processes or compositions.

Having described the invention, what is claimed is:

1. A process for preparing, in dry pulverulent form, a fatty acid sarcosinate, the acyl radical of which contains from 10 to 18 carbon atoms, which comprises: atomizing a pumpable aqueous solution of greater than 10 percent by weight of said sarcosinate, at a temperature below that at which the sarcosinate decomposes under the processing conditions, into heated drying gas at the top of a vertical zone through which the atomized material falls; admitting cooling air into said zone below the point of atomization so directed as to pursue a path about the suspension of atomized particles in drying gas, thereby cooling the zone boundary to a temperature below that of the drying gas while also inhibiting contact of particles with the zone boundary and lowering the temperature of any particles which do pass through the cooling air, so as to decrease the tendency of fatty acid sarcosinate to melt and accumulate on the zone boundary; and withdrawing from the zone at the bottom thereof dry rigidified fatty acid sarcosinate particles and a mixture of spent drying gas and cooling air.

2. The process of claim 1 in which additional cooling air is introduced at the bottom of the vertical zone to aid in cooling and rigidifying the dried fatty acid sarcosinate particles by direct contact therewith.

3. A process for preparing, in dry pulverulent form, an alkali metal acid sarcosinate, the fatty acyl radical of which is saturated and contains from 12 to 16 carbon atoms, which comprises: atomizing a pumpable aqueous solution of 10 to 40 percent by weight of said sarcosinate, at a temperature between 30° F. and 140° F. at which the sarcosinate is stable under the processing conditions, into heated drying gas at a temperature between 250° F. and 600° F. at the top of a vertical zone through which the atomized material falls; admitting cooling air below 100° F. into said zone so directed as to pursue a path about the suspension of atomized particles in drying gas, thereby cooling the zone boundary to a temperature below that of the drying gas while also inhibiting contact of particles with the zone boundary and lowering temperature of any particles which do pass through the cooling air so as to decrease the tendency of fatty acid sarcosinate to melt and accumulate on the zone boundary; and withdrawing from the zone at the bottom thereof dried rigidified alkali metal fatty acid sarcosinate particles and a mixture of spent drying gas and cooling air, said particles of fatty acid sarcosinate being at a temperature lower than that at which the sarcosinate is tacky, and the mixture of spent drying gas and cooling air withdrawn being at a temperature between 100° F. and 250° F.

4. A process for preparing in dry pulverulent form an alkali metal fatty acid sarcosinate, the fatty acyl radical of which is saturated and contains from 12 to 16 carbon atoms, which comprises: atomizing a pumpable aqueous solution of 10 to 40 percent by weight of said sarcosinate, containing no more than 10 percent by weight, on a sarcosinate basis, of fatty acid material, at a temperature between 70° F. and 140° F. at which the sarcosinate is stable under the processing conditions, into heated drying gas at a temperature between 375° F. and 500° F. at the top of a vertical zone through which the atomized material falls; admitting cooling air below 100° F. tangentially to said zone so directed as to pursue a path about the suspension of atomized particles in drying gas, thereby cooling the zone boundary to a temperature below that of the drying gas while also inhibiting contact of particles with the zone boundary and lowering the temperature of any particles which do pass through the cooling air so as to decrease the tendency of fatty acid sarcosinate to melt and accumulate on the zone boundary; introducing additional cooling air below 100° F. at the bottom of the vertical zone to aid in cooling and rigidifying the dried alkali metal fatty acid sarcosinate particles by direct contact therewith; and withdrawing from the zone at the bottom thereof dried rigidified fatty acid sarcosinate particles and a mixture of spent drying gas

and cooling air between 100° F. and 190° F., said particles of fatty acid sarcosinate being at a temperature below about 200° F. and lower than that temperature at which they are tacky.

5. A process for preparing in dry pulverulent form an alkali metal fatty acid sarcosinate, the fatty acyl radical of which is saturated and contains from 12 to 16 carbon atoms, which comprises: atomizing a pumpable aqueous solution of 10 to 40 percent by weight of said sarcosinate, containing no more than 10 percent by weight, on a sarcosinate basis, of fatty acid material, at a temperature between 70° F. and 140° F. at which the sarcosinate is stable under the processing conditions, into heated drying gas at a temperature between 375° F. and 500° F. at the top of a vertical zone through which the atomized material falls, the atomization being such as to result in droplets of such size that on drying they will become particles, the diameters of which will be substantially in the 10–150 micron range; admitting cooling air below 100° F. tangentially to said zone so directed as to pursue a path about the suspension of atomized particles in drying gas, thereby cooling the zone boundary to a temperature below that of the drying gas while also inhibiting contact of the particles with the zone boundary and lowering the temperature of any particles which do pass through the cooling air so as to decrease the tendency of fatty acid sarcosinate to melt and accumulate on the zone boundary; introducing additional cooling air below 100° F. at the bottom of the vertical zone to aid in cooling and rigidifying the dried fatty acid sarcosinate particles by direct contact therewith; and withdrawing from the zone at the bottom thereof dried rigidified alkali metal fatty acid sarcosinate particles of at least 80 percent alkali metal fatty acid sarcosinate, less than about 12 percent moisture and less than about 8 percent fatty acid material, said particles being in hollow substantially spherical bead form of diameters substantially within the range of 10–150 microns and at a temperature below 200° F. and lower than that temperature at which the sarcosinate is tacky, and a mixture of spent drying gas and cooling air between 100° F. and 190° F.

6. A process for preparing in dry pulverulent form sodium N-lauroyl sarcosinate which comprises: atomizing a pumpable aqueous solution of 30 to 40 percent by weight of said sarcosinate, containing no more than 10 percent by weight, on a sarcosinate basis, of fatty acid material, at a temperature between 70° F. and 140° F. at which the sarcosinate is stable under the processing conditions, into heated drying gas at a temperature between 375° F. and 500° F. at the top of a vertical zone through which the atomized material falls; admitting cooling air below 100° F. tangentially to said zone so directed as to pursue a path about the suspension of atomized particles in drying gas, thereby cooling the zone boundary to a temperature below that of the drying gas while also inhibiting contact of particles with the zone boundary and lowering the temperature of any particles which do pass through the cooling air so as to decrease the tendency of fatty acid sarcosinate to melt and accumulate on the zone boundary; introducing additional cooling air at 100° F. at the bottom of the vertical zone to aid in cooling and rigidifying the dried fatty acid sarcosinate particles by direct contact therewith; and withdrawing from the zone at the bottom thereof dried rigidified fatty acid sarcosinate particles and a mixture of spent drying gas and cooling air at a temperature between 100° F. and 190° F., said particles of fatty acid sarcosinate being at a temperature below 200° F. and lower than that temperature at which the sarcosinate is tacky.

References Cited in the file of this patent

UNITED STATES PATENTS

1,652,900	Lamont	Dec. 13, 1923
1,779,517	Stevenson et al.	Oct. 28, 1930
2,689,170	King	Sept. 14, 1954

OTHER REFERENCES

Perry: Chemical Engineers' Handbook, 3rd ed., copyright 1950, pp. 838–846.

Brown et al.: United Operations (1950), John Wiley & Sons, Inc., New York, New York, pp. 562–564.