Oct. 13, 1964 J. A. MONICK ETAL 3,152,947

PROCESS FOR DRYING HIGHER FATTY ACID SARCOSINES

Original Filed July 7, 1958 2 Sheets-Sheet 1

Fig. 2.

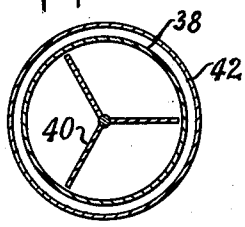


Fig. 1.

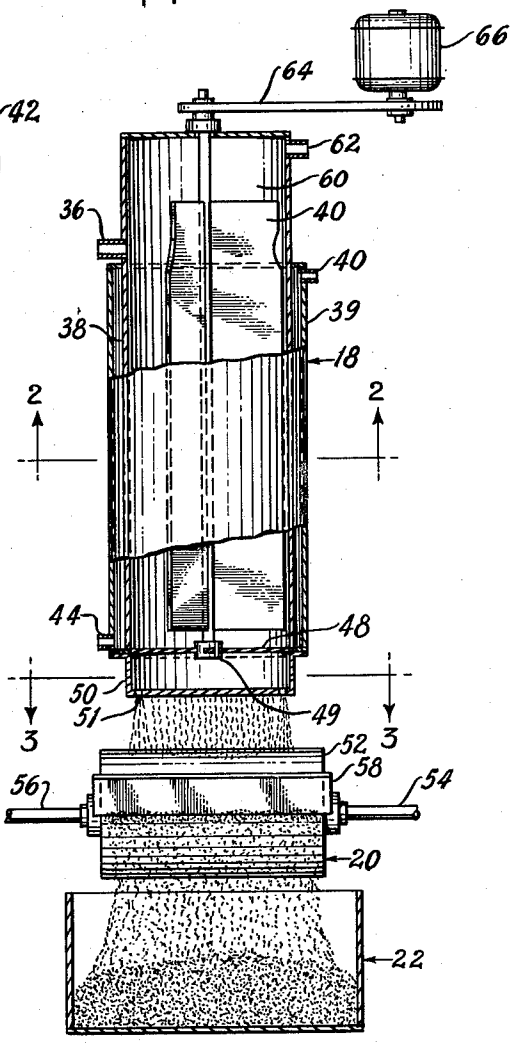
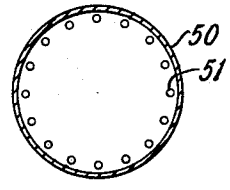


Fig. 3.



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Original Filed July 7, 1958 2 Sheets-Sheet 2

FIG. 4.

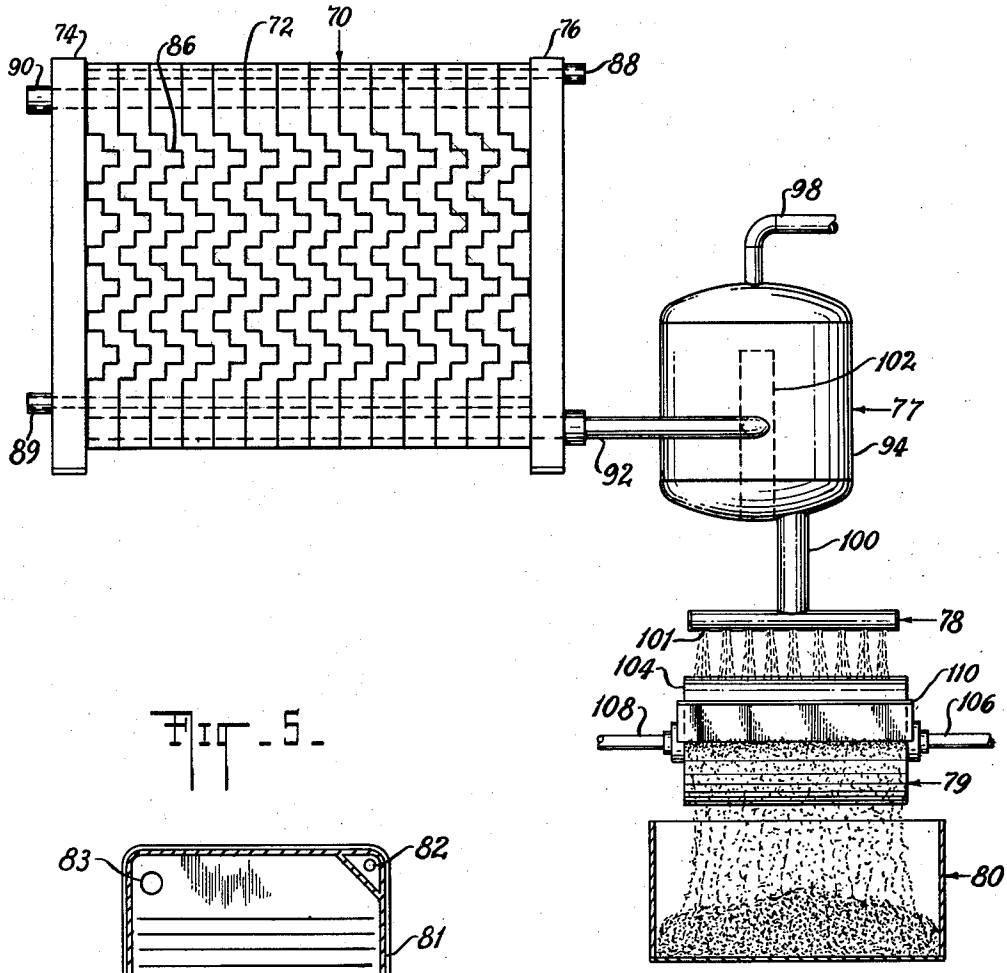
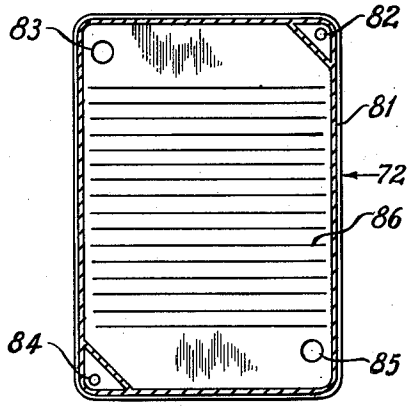


FIG. 5.



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PROCESS FOR DRYING HIGHER FATTY ACID SARCOSINES

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Continuation of abandoned application Ser. No. 746,763, July 7, 1958. This application Sept. 18, 1962, Ser. No. 225,408

12 Claims. (Cl. 159—49)

The present invention relates to a process for heat drying aqueous solutions of higher fatty acid sarcosines and salts thereof.

Higher fatty acid sarcosines are useful as constituents of dental preparations such as tooth pastes and powders, in detergent bars, and like compositions.

For a number of such uses, e.g. incorporation in tooth powders and dry detergent compositions, it is desirable that the higher fatty acid sarcosine be available in dry solid form, preferably a particulate solid form. The fatty acid sarcosines and their salts are more stable toward chemical and biological decomposition when in dry solid form than when in the form of an aqueous solution. Also, when the product must be transported shipping costs are lower. Inasmuch as the usual manufacturing process produces the higher fatty acid sarcosines in aqueous solution, a drying step is necessitated. However, due to the peculiar properties of the higher fatty acid sarcosines and concentrated solutions thereof, the drying of such solutions presents a troublesome problem.

Higher fatty acid sarcosines are difficult to dry satisfactorily by conventional methods because of their tendency to decompose on exposure to elevated temperatures for an extended period of time. The higher fatty acid sarcosines are also characterized by a tendency to gel when in high concentrations in aqueous solutions. Such gels are characterized by very poor heat and mass transfer rates, and are very difficult to dry without experiencing thermal decomposition of the sarcosine.

It has previously been proposed to overcome these problems by spray drying higher fatty acid sarcosines according to a process in which a solution of the sarcosine in water is atomized into a heated drying gas at the top of a vertically elongated zone, while cooling air is admitted to the zone at a point below that of atomization, to rigidify the dried sarcosinate particles. The dried fatty acid sarcosine particles are withdrawn at the bottom of the zone as is a mixture of spent drying gas and cooling air.

A process for heat drying aqueous higher fatty acid sarcosines and their salts has now been discovered which is characterized by advantages of reduced thermal decomposition during processing, substantially quantitative recovery of fatty acid sarcosine solids (e.g. greater than 99%) and the production of dust-free dry products. Negligible increase in free fatty acid content is experienced in the course of drying higher fatty acid sarcosines in accordance with the present procedure, thus facilitating preparation of higher fatty acid sarcosines containing less than 15% free fatty acid (or fatty acid soap) which is preferred for use in dentifrices.

In accordance with the present invention a continuous process for heat drying a higher fatty acid sarcosine comprises forming a thin layer of a fluid aqueous solution of a higher fatty acid sarcosine, heating said layer to a temperature sufficient to boil off the water therein and melt said higher fatty acid sarcosine for a time sufficient to substantially dry said higher fatty acid sarcosine and melt it, and cooling said dry molten fatty acid sarcosine to solid condition.

The normally solid sarcosines contemplated as within the present invention are amides formed from sarcosine and a higher fatty acid or mixtures thereof. These fatty acids are straight chain higher mono-carboxylic acids containing from 10 to 18 carbon atoms and may be saturated or unsaturated as long as the sarcosine derivative or salt thereof is normally solid. Examples of such fatty acids are lauric acid, myristic acid, palmitic acid, oleic acid, coconut oil fatty acids, and hydrogenated tallow fatty acids, it being especially preferred to carry out the present process employing those fatty acid sarcosines which either because of their relatively low stability, low melting point or tendency to gel, or combination of such characteristics, cannot be dried in conventional ways into discrete, non-dusty particles. Examples of sarcosines which readily form gels when in high concentration in aqueous solution are those higher fatty acid sarcosines prepared from straight chain saturated fatty acids containing 12 to 16 carbon atoms, and especially the sodium salts thereof.

The sarcosines of the present invention may be in the acid form or may be employed as a salt (sarcosinate) and the term "higher fatty acid sarcosine" as used herein and in the appended claims is intended to encompass within its scope both the acid and its salts. It is preferred to employ salts selected from those formed by alkali metals or nitrogen-containing radicals. More particularly they may be sodium, potassium, ammonium, amine, mono-, di-, or tri-ethanolamine salts. It is particularly preferred to carry out the present process employing the sodium salts of sarcosinates whose fatty acyl groups are saturated and contain from 12 to 16 carbon atoms.

The present higher fatty acid sarcosines 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, trimethyl amine. The resulting amide is separated, acid washed and subsequently, if desired, neutralized with a base containing a desired cation. Ordinarily the sarcosine or sarcosinate is obtained in solution, and to promote economical drying of such a solution the concentration of higher fatty acid sarcosine in the solution should be as high as possible, at least 10%. Since at high concentrations, e.g. above about 40–45% in water in the cases of sodium N-lauroyl sarcosinate and sodium N-palmitoyl sarcosinate, a gel is formed at room temperature and above, such a gelation concentration is the upper feasible solution concentration limit of the aqueous solutions dried in carrying out the present process. The addition of solvents such as ethanol and isopropanol is within the scope of the present invention as these solvents are removed along with the water in the course of the present process. Inasmuch as formation of the molten higher fatty acid sarcosine is an essential feature of the present process, it is apparent that the higher fatty acid sarcosine employed should be relatively pure and substantially free of materials having a higher melting point such as, for instance, inorganic salts, e.g. sodium sulfate and sodium chloride. Thus the feedstock employed in the present process normally will be a non-gelled, fluid aqueous solution of a higher fatty acid sarcosine substantially pure and containing from 10 to 40% total solids.

The word "solution" is used herein to describe fluid aqueous preparations including true solutions, suspensions, collodial suspensions, dispersions, and slurries, but is exclusive of gels. These aqueous solutions may contain other liquid ingredients such as low molecular weight alcohols, e.g. ethanol and isopropanol which serve to diminish solution viscostiy and to increase the extent of fatty acid sarcosinate solubilty without gel formation,

and liquid preservative agents which may be employed to protect the higher fatty acid sarcosine from attack by bacteria or microorganisms. The higher fatty acid sarcosine solution may also contain adjuvant materials such as fillers, additives or impurities which do not significantly detract from the utility of the product or seriously interfere with the present process, however, when intended for used in dental preparations for its acid inhibiting effect the sarcosine should be as free as possible of fatty acids and fatty acid soaps because of the deactivating properties thereof.

In carrying out the instant drying procedure, the fluid aqueous higher fatty acid sarcosine, preheated if desired but normally at a temperature below about 100° C., is disposed as a thin moving layer of liquid from which solvent is vaporized as the layer progresses through a heating zone. The layer employed is preferably disposed on a heated metallic surface and is less than ¼ inch thick, preferably being a thin film having a thickness of less than 1/16 inch, typically between 1/100 and 1/16 of an inch. In the previously mentioned heated zone the layer of higher fatty acid sarcosine is heated to a temperature which is above the boiling point of the solvent from the aqueous layer and above the melting point of the higher fatty acid sarcosine solid, typically to a temperature on the order of 145–195° C., in the case of the salts, and 105–145° C. in the case of the lower melting free acids.

As the layer progresses through the heated zone it is maintained at a temperature above its boiling point for a period of time sufficient for substantially all of the solvent therein to evaporate or boil off, and to form substantially dry molten fatty acid sarcosine, but insufficient to cause any significant pyrolytic decomposition of the higher fatty acid sarcosine. Typically, such a period of time of exposure to elevated temperature is on the order of less than 2 minutes, and preferably less than 30 seconds, typically being on the order to 10 to 30 seconds. The molten higher fatty acid sarcosine is then removed from the heated zone and immediately is cooled to a stable solid condition to form a rigid solid product containing less than about 6%, and preferably less than about 2% moisture as at moisture levels of about 6.5% and above, non-rigid tacky gels tend to form.

The thin layer of higher fatty acid sarcosine which is processed in accordance with the present invention is agitated throughout the boiling period. While suitable turbulence may be obtained by means of turbulent fluid flow, it is much preferred to carry out the instant process employing mechanical agitation of a thin film of liquid being heated on a metallic surface.

The dry molten fatty acid sarcosine may be cooled in any suitable manner to form flakes, pellets, ribbons, powder, granules, spheres, strips, bulk form, or the like. It is preferred to form dense, non-dusty readily soluble solid pellets of maximum diameter less than about ⅜ inch by forming the molten fatty acid sarcosine into discrete liquid droplets which are solidified by spray cooling, immersion in a cool, non-solvent liquid, or deposition on a chilled surface, e.g. a cooling roll. Such dense particulate solids have a high bulk density and are economical to ship, and are readily wetted by water, dissolving easily without floating or foaming.

In order to indicate more specifically the advantages and features of the novel process of the present invention, the following examples are set forth, which taken in conjunction with the accompanying drawings, illustrate preferred form of apparatus for carrying out the present invention. In these examples all parts and percentages are by weight unless otherwise specified.

Example I

FIG. 1 shows diagrammatically the various comopnent parts of a preferred apparatus for carrying out the process of the present invention.

FIG. 2 is a section taken on line 2—2 of FIG. 1; and FIG. 3 is a section taken on line 3—3 of FIG. 1.

The apparatus of FIGS. 1–3 comprises a wiped film evaporator 18, a cooler 20, and a product collection bin 22. The wiped film evaporator comprises essentially a closed vertically elongated tube 38 containing a concentrically mounted rapidly rotating vaned agitator 40 which forces the liquid content of the evaporator against the inner wall of said vertical elongated tube 38 thus spreading the liquid in a thin film and maintaining the liquid in rapid movement so as to insure intimate mixing, heat transfer and prevent overheating. A feed supply nozzle 36 is located in the upper portion of the tube 38. The portion of the vertical tube 38 below the feed point 36 of the evaporator 18 is provided with a jacket 39 through which a heating medium is passed, the heating medium entering and leaving said jacket by means of taps 44 and 46 at the base and top thereof respectively. The liquid content of the evaporator flows down the inside surface of the tube 38, through a spider 48 which carries a foot bearing 49 supporting the vaned agitator 40, and collects in a closed cylindrical distributor 50 which is provided with perforations 51 near the outer edge of the base thereof. The distributor is steam traced (not shown) in order to prevent cooling and solidification of the material therein.

Liquids passing through the perforations fall as droplets on a rotating drum 52 which is chilled by cooling water supplied to the interior thereof through hollow trunnions 54 and 56. Material is removed from the surface of the cooling roll by a doctor blade 58, and falls into the product collection bin 22.

The portion of the vertical elongated tube 38 above the feed point 36 forms a separation chamber 60 for recovery of liquid entrained in the vapors being removed. A conduit 62, which communicates with the separation chamber 60, is connected to a source of slight vacuum (on the order of 4 millimeters of water) so as to facilitate removal of solvent vapors from the reactor 18. The vaned agitator 40 is rotated by a belt 64 driven by an electric motor 66.

In operation a 36% fluid aqueous solution of (99% sodium N-lauroyl sarcosinate and 1% lauric acid soap) solids is fed at room temperature of 25° C. to the evaporator through the feed conduit 36 at a rate of 130 pounds of solution per hour for a 4-hour period. Rapid rotation of the agitator spreads and turbulently agitates the solution as a thin film on the interior of wall 38 of the evaporator, the clearance between the periphery of the vanes 40 and wall 38 and the thickness of the film being approximately 1/32 inch. Saturated steam at 150 p.s.i.g. (185° C.) is supplied to the jacket through tap 46, and condensate is removed at tap 44.

Vapors at a temperature of 102° C. are removed through conduit 62 by application thereto of a vacuum of 4 millimeters of water. The vaporizing surface in the evaporator is 9 square feet, and the agitator runs at 900 r.p.m., corresponding to a peripheral velocity of 2,820 feet per minute at the outer edges of the vanes. Molten, substantially dry sodium N-lauroyl sarcosinate issues from the base of the evaporator and falls into the distributor head 50 from which it issues as droplets through the holes 51 and falls onto the cooling roll 52, the time of passage from the feed conduit to the cooling roll being approximately 22 seconds. The holes in the distributor are made by a No. 30 drill, and the pellets which form on the cooling roll are flattened droplets approximately 1/16 inch thick and ¼ of an inch in diameter. The pellets are removed from the cooling roll at approximately 25° C. by the doctor blade 58 and fall into the collection bin 22.

Analysis of the product obtained in the collection bin discloses that there has been no increase in the soap content during processing. The product contains 97.6% sodium N-lauroyl sarcosinate, 1.0% lauric acid soap, and

only about 1.4% moisture. The pellets form a non-tacky, free-flowing, dust-free particulate product having an apparent density of about 0.55 gram/cc. The evaporate is substantially free of any sarcosinate solids, the extent of recovery of fatty acid sarcosinate in the product bin being approximately 99.8% of that fed to the evaporator.

Similar results may be obtained employing a wiped film evaporator having a frusto-conical or tapered shape, and the evaporator employed may be disposed in any suitable plane, e.g. vertical or horizontal.

Example II

The procedure of Example I is repeated employing a fluid feed solution containing 39.6% sodium N-lauroyl sarcosinate, 0.4% sodium laurate, 40% water, and 20% ethanol at room temperature. The feed rate is 30 pounds per hour per square foot of heated surface in the evaporator, and the evaporator is heated with saturated steam at about 145° C. No increase in soap content is experienced, the product being solid, free-flowing, non-tacky, rigid, alcohol-free particles which contain 93.25% sodium N-lauroyl sarcosinate, 5.75% moisture, and 1.0% soap.

Example III

The procedure of Example II is repeated employing, as a feedstock, a fluid mixture containing 85% molten N-lauroyl sarcosine (melting point about 45° C.) and 15% water. The feed rate is 20 pounds per hour per square foot of heated surface, and the temperature of the feed is about 75° C. The saturated steam used is at about 127° C. The product obtained contains 98.2% N-lauroyl sarcosine, 1.5% free fatty acid, and 0.3% moisture.

Example IV

The procedure of Example II is repeated employing, as a feedstock, a fluid solution of 16.22% sodium N-palmitoyl sarcosinate (melting point 147° C.) and 0.78% sodium palmitate in water at room temperature. The feed rate is 30 pounds per hour per square foot of heated surface in the evaporator, and the evaporator is heated with saturated steam at about 185° C. Solid, free-flowing, non-tacky, rigid pellets are produced which contain 93.48% sodium N-palmitoyl sarcosinate, 4.52% sodium palmitate, and 2% moisture, the proportion of soap present being substantially the same as in the feed.

Example V

FIG. 4 shows diagrammatically the various components of an alternate apparatus for carrying out the process of the present invention. It comprises a commercially available thin film heater **70** constructed in a manner similar to a conventional plate and frame filter press in that it consists of a plurality of thin corrugated rectangular sheets **72** clamped between two end plates **74, 76** (by means not shown), a centrifugal liquid-vapor separator **77**, a liquid distributor **78**, a product cooler **79**, and a collection bin **80**.

FIG. 5 illustrates a typical construction of one of the sheets **72**. The sheets are spaced and sealed by gaskets **81** around their outer edges and are provided, in a manner similar to the plates of a conventional plate and frame filter press, with openings **82, 83, 84** and **85** in each corner for flow of fluid. These openings may be communicated with or sealed off from the space between any pair of adjacent plates by virtue of the shape of the gaskets **81** employed. Thus in the embodiment illustrated in FIG. 5, openings **83** and **85** communicate with the illustrated surface of the plate **72**, and openings **82** and **84** do not. In use, the plates are gasketed so as to cause the heating medium to pass through every second successive space between pairs of plates and fluid being heated to pass through each alternate space.

Corrugations **86** on the plates cause considerable turbulence of the fluid flowing thereon because of the frequent changes in direction and cross sectional area of the space between surfaces of adjacent plates. The plate to plate spacing through which the fluid flows averages from $\frac{1}{16}$ to $\frac{7}{32}$ of an inch, thus providing, in the space between each pair of plates, for the formation of a turbulently flowing stream consisting, in essence, of two liquid layers each from $\frac{1}{32}$ to $\frac{7}{64}$ of an inch thick.

The heater of FIG. 4 is provided with a conduit **88** through which heating medium is supplied to the plates and a conduit **89** for exhausting the used heating medium. A conduit **90** supplies feed to be heated to the spaces between the plates, and a conduit **92** is provided for removal of the heated product.

The product conduit **92** is tangentially connected into the periphery of a centrifugal separator **77** comprising a closed cylindrical vessel **94** provided with an overhead line **98** for removal of vapors and a drain conduit **100** for removal of liquids. A center post **102** is provided to maintain regular circulation of materials in the separator and to prevent turbulence.

The drain conduit **100** supplies the distribution head **78**. Perforations **101** in the bottom of the distribution head dispense droplets onto the cooler **79**. In use, the separator **77**, the drain conduit **100**, and the distribution head **78** are steam traced (not shown) to insure that the materials therein are maintained in molten condition.

The cooler **79** comprises a cooling roll **104** which is chilled by means of cold water supplied thereto through hollow trunnions **106** and **108**. Cooled material is removed from the surface of the cooling roll by a doctor blade **110** and falls into the collection bin **80**.

In operation a 38% aqueous solution of 98.5% sodium N-lauroyl sarcosinate and 1.5% sodium laurate is fed to the heater through feed conduit **90** at a rate of approximately 0.96 pound of said sarcosinate solids per hour per square foot of heating surface. The feed is supplied at a gauge pressure of about 95 pounds per square inch and is passed through every second space between plates. Saturated steam at a temperature of 170° C. is employed as a heating medium and is passed through the space between alternate pairs of plates. The solution boils in the heater and forms a mixture of steam and molten sodium N-lauroyl sarcosinate which issues from the heater through conduit **92** and passes into the centrifugal separator **77**. Water vapors are removed from the separator through the overhead line **98** by the application thereto of a vacuum of 4 millimeters of water, and liquid sodium N-lauroyl sarcosinate drains from the base of the separator through the drain conduit **100** into the distributor **78**. Droplets of sodium N-lauroyl sarcosinate issue from the distributor, fall onto the cooling roll **104** where they are immediately cooled below their solidification point and form small solid pellets, and are removed from the roll by the doctor blade **110** and collected in the pan **80**. The total time of passage from the feed conduit **90** to the cooler **79** is on the order of less than 45 seconds.

The pellets produced contain 95% sodium N-lauroyl sarcosinate, 2.5% lauric acid soap, and 2.5% moisture.

The effectiveness of heating the material being dried to a temperature above its melting point, as in the foregoing examples, is illustrated by simulating the procedure of Example I using as a heating medium, saturated steam at a temperature of only 121° C. A feed rate of 22 pounds per hour per square foot of heated surface is employed. The solution concentrates only to about 40% solids, and then, as it approaches the gel condition, becomes so viscous that it cannot be removed from the base of the evaporator. The advantages of the present process which readily produces a free-flowing, substantially dry molten product are apparent.

This applicaiton is a continuation of our pending application S.N. 746,763, filed July 7, 1958, now abandoned.

What is claimed is:

1. A continuous process for heat drying of fluid aqueous solution of a normally rigid solid higher fatty acid sarcosinate which comprises moving such solution turbu-

lently over a heated metallic surface in direct contact therewith, heating said solution thereby to a temperature above both the boiling point of the solution and the melting point of the sarcosinate, vaporizing substantially all of the aqueous solvent from said solution and melting the substantially dry sarcosinate thus formed before contact with any substantial amount of gas other than said vapor, forming the molten dried sarcosinate into discrete liquid droplets, and cooling said droplets to rigid solid pellets of dry sarcosinate.

2. A containuous process for heat drying of fluid aqueous solution of a normally solid rigid higher fatty acid sarcosinate as set forth in claim 1 in which the solution is moved turbulently as a wiped layer.

3. A continuous process for heat drying of fluid aqueous solution of a normally rigid solid higher fatty acid sarcosinate as set forth in claim 1 in which the solution is caused to move turbulently by frequent changes in cross sectional area as it flows between adjacent heating areas of said metallic surface.

4. A continuous process for converting an aqueous solution of sodium N-lauroyl sarcosinate at a solids concentration within the range of about 10% to 40% into solid pellets having a moisture content not exceeding about 6% as set forth in claim 3 in which said stream is a thin wiped cylindrical layer and the vapor is separated countercurrently to said moving stream of solution as it is formed.

5. A continuous process for converting an aqueous solution of sodium N-lauroyl sarcosinate at a solids concentration within the range of about 10% to 40% into solid pellets having a moisture content not exceeding about 6% as set forth in claim 3 in which said stream frequently changes in direction and cross-section and the vapor and dried molten sarcosinate move concurrently until they are separated and the separation takes place by centrifugal action.

6. A continuous process for converting an aqueous solution of sodium N-lauroyl sarcosinate at a solids concentration within the range of about 10% to 40% into solid pellets having a moisture content not exceeding about 6% which comprises moving a stream of said solution continuously through a passageway, agitating and rapidly heating said solution as it moves through said passageway to a temperature within the range of about 145° to 195° C. for a period on the order of less than 2 minutes whereby aqueous solvent is converted into vapor until not more than 6% moisture is present in the sarcosinate and the thus dried sarcosinate is in molten condition, separating said dried molten sarcosinate from said vapor before contact with any substantial amount of gas other than said vapor, forming said separated dried molten sarcosinate into discrete droplets, and cooling said droplets to solid pellets of sodium N-lauroyl sarcosinate containing not over 6% moisture.

7. A continuous process for heat drying, with minimum thermal decomposition, an aqueous higher fatty acid sarcosinate salt which is normally a rigid solid comprising forming a flowable non-gelled aqueous solution of a normally solid higher fatty acid sarcosinate salt having a tendency to gel when in aqueous solution at high concentration, disposing said flowable sarcosinate solution as a wiped liquid film, turbulently agitating and rapidly heating said film of sarcosinate solution to a temperature above both the boiling point of the solution forming said film and the melting point of said higher fatty acid sarcosinate salt, vaporizing substantially all of the water in said film to produce said higher fatty acid sarcosinate in molten, substantially dry form, forming droplets of said sarcosinate and immediately cooling said droplets to a temperature below its melting point to form higher fatty acid sarcosinate salt in the form of rigid, substantially dry, non-tacky pellets substantially free of any thermal degradation product.

8. A continuous process for heat drying an aqueous higher fatty acid sarcosinate as set forth in claim 7 wherein said sarcosinate is sodium N-lauroyl sarcosinate.

9. A continuous process for heat drying, with minimum decomposition, an aqueous higher fatty acid sarcosinate which is normally a rigid solid comprising forming, at a temperature below 100° C., an aqueous solution containing from about 10 to 40% by weight of a higher fatty acid sarcosinate, said fatty acid being a straight chain monocarboxylic acid containing from 12 to 16 carbon atoms, disposing said solution as a wiped liquid film less than 1/16 inch thick on a heated metallic surface, turbulently agitating and rapidly heating said film to a temperature above the melting point of said sarcosinate for a period of from 10 to 30 seconds sufficient to boil off substantially all of the water therein and produce said higher fatty acid sarcosinate in molten, substantially dry form, forming droplets of said sarcosinate and immediately cooling said droplets to a temperature below about 40° C. to produce higher fatty acid sarcosinate in the form of rigid, substantially dry, non-tacky pellets substantially free of any pyrolytic degradation product.

10. A continuous process for heat drying an aqueous solution of a normally rigid solid higher fatty acid sarcosinate which comprises moving turbulently a wiped layer of a fluid aqueous solution thereof over a heated surface, heating said layer to a temperature above both the boiling point of said solution and the melting point of said fatty acid sarcosinate, vaporizing substantially all of the water in said layer and melting the substantially dry fatty acid sarcosinate thus formed, forming said molten fatty acid sarcosinate into discrete liquid droplets, and cooling said droplets to rigid solid pellets of dry, higher fatty acid sarcosinate.

11. A continuous process for heat drying an aqueous solution of a higher fatty acid sarcosinate as set forth in claim 10 wherein said higher fatty acid sarcosinate is sodium N-lauroyl sarcosinate.

12. A continuous process for heat drying an aqueous solution of a higher fatty acid sarcosinate as set forth in claim 10 wherein said higher fatty acid sarcosinate is sodium N-palmitoyl sarcosinate.

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