Nov. 5, 1957 R. F. HEALD ET AL 2,812,018

PROCESS FOR DRYING SYNTHETIC ORGANIC DETERGENT COMPOSITIONS AND THE LIKE

Filed March 2, 1953

Fig. 1.

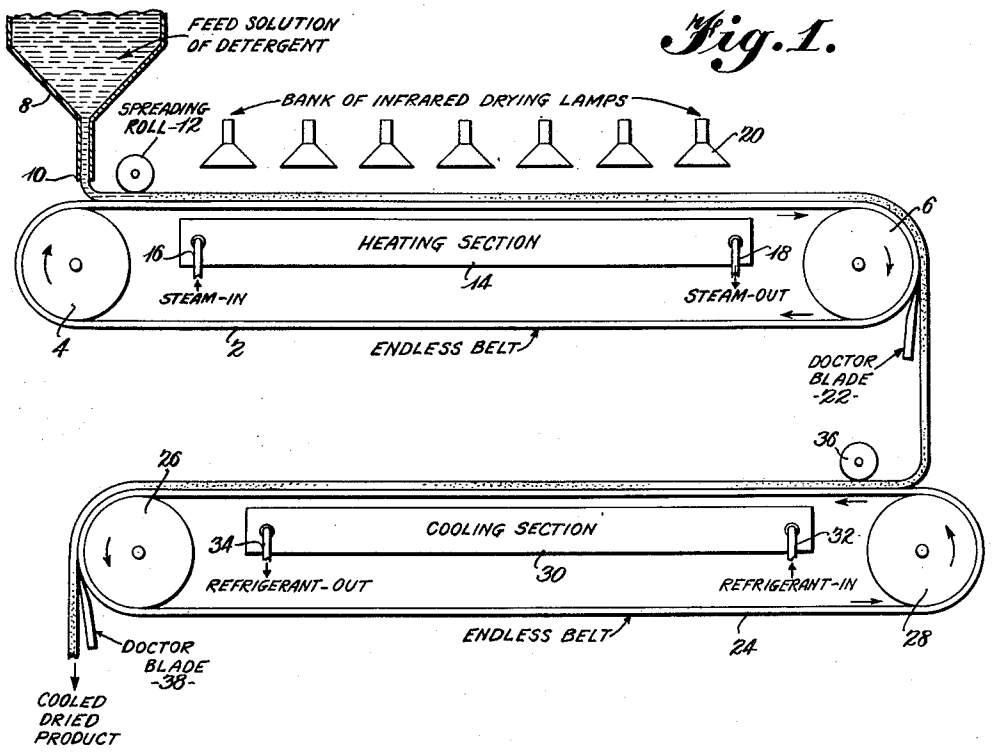


Fig. 2.

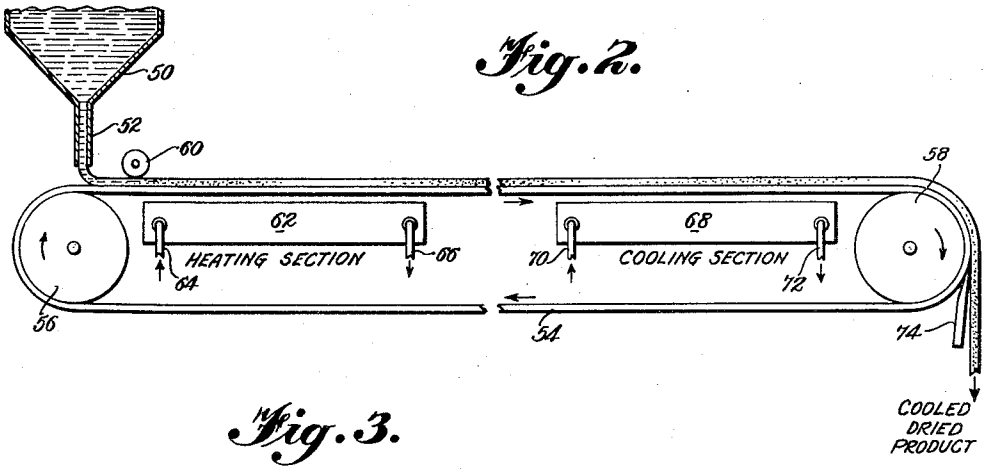
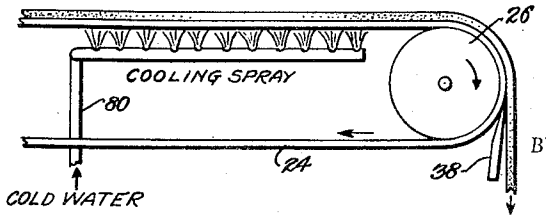


Fig. 3.



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United States Patent Office 2,812,018 Patented Nov. 5, 1957

2,812,018

PROCESS FOR DRYING SYNTHETIC ORGANIC DETERGENT COMPOSITIONS AND THE LIKE

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Application March 2, 1953, Serial No. 339,578

3 Claims. (Cl. 159—49)

This invention relates to the drying of synthetic organic detergent compositions and the like, and more particularly to the drying and cooling of synthetic organic detergent compositions such as of the sulphated monoglyceride type, especially when in salt-free form.

One of the principal objects of the present invention is to provide a new and improved method for preparing in cool dry form synthetic organic detergent compositions and the like. A second object of the invention is to provide a method for drying synthetic organic detergent compositions, especially when in the salt-free form, without decomposition or contamination. A third object of the invention is to provide a method for drying salt-free synthetic organic detergent compositions having a tendency to melt at the drying temperature under temperature conditions above their melting point, and then cooling suddenly so that the dried product can be maintained in suitable form for further processing. A fourth object of the invention is to provide a method of drying salt-free synthetic organic detergent compositions which will be economical in operation, generally productive of a superior product, and substantially free of decomposition products that might otherwise be obtained as a result of subjecting such materials to relatively high temperatures for drying purposes if processed in accordance with methods heretofore known in the art.

The manner in which these and other objects and features of the invention are attained will appear more fully from the following description thereof, in which reference is made to typical and preferred procedures in order to indicate more clearly the nature of the invention, but without intending to limit the invention thereby.

Synthetic organic detergent compositions of the type to which the present invention is particularly adaptable include for example alkali metal salts such as sodium salts of sulphated monoglycerides of higher fatty acids having from 8 to 24 carbon atoms such as are derived from vegetable and animal oils, such as coconut oil, hydrogenated tallow fatty acids and the like. The invention is especially applicable to salt-free compositions of the foregoing type, i. e., compositions that are free or substantially free of inorganic salts such as sodium sulphate and the like.

Such compositions are well known in the art and may be prepared in any suitable manner. According to one known method, the compositions may be formed by the interaction of a fatty oil, anhydrous glycerine and fuming sulphuric acid, preferably in the approximate proportion of 1 molecular weight of fatty oil to 2 molecular weights of glycerine and in excess of 3 molecular weights of sulphuric acid. The resultant acid mixture then is neutralized with caustic soda or other alkaline neutralizing agent. The inorganic salt present in the product produced by this method may be removed, for example, by alcohol extraction whereupon the sodium or other alkali metal salts of the sulphated monoglycerides are

obtained in the desired substantially inorganic-salt-free condition.

These detergent compositions are frequently recovered in the form of aqueous-alcoholic solutions of from about 20 to 50% solids content, say about 40% solids content, and it is desirable thereafter to dry them to a moisture content of about 3% or less in order to place them in a form better suited for certain uses, e. g., in preparing dental detergent compositions.

Various methods for reducing the moisture content of such compositions are already known in the art, for example, spray drying and the like, but unfortunately such prior art methods are attended by certain difficulties which it is the purpose of the present invention to avoid. For example, in the course of spray drying thermosensitive organic materials such as these, there may be difficulty in overcoming their tendency to oxidize and/or hydrolyze to undesired by-products. Furthermore, it is particularly difficult to eliminate troubles due to contamination by dirt which, as is well known, is an important problem confronting the spray-drying engineer. The foregoing and other difficulties are overcome, and other advantages are obtained, by the process of the present invention.

The present invention is based upon the discovery that solutions in suitable solvents, for example, water, alcohol, or mixtures thereof, of detergents of the type described herein may be very conveniently and satisfactorily dried to any desired low moisture content and thereafter cooled and collected by employing a drying and cooling surface in the form of a continuously moving belt. The belt preferably is fabricated of Inconel or stainless steel or other suitable corrosion-resistant material having good heat transfer properties.

According to a preferred embodiment of the invention, the solution containing the synthetic organic detergent is applied to the upper surface of a continuously moving belt where it may be spread out into a relatively thin film by knife or spreading roll. The moving belt carrying the thin film of synthetic organic detergent solution then passes through a heating zone where the film is subjected to heat applied above and/or below the belt surface at such a rate as to drive off substantially all or at least a predetermined portion of the solvent, after which the hot, dried, or substantially dried, solid film of detergent is passed through a cooling zone either on the same or a different belt surface where it is rapidly cooled to substantially room temperature or lower if desired. In this fashion, the synthetic organic detergent solution is reduced from an initial solids content of e. g., approximately 40% to a dry or substantially dry product having the desired moisture content, for example, about 3% or less by weight.

In order still further to illustrate the present invention, reference is made to the accompanying drawing in which Figure 1 shows in somewhat diagrammatic fashion an apparatus in which two separate endless belts are employed;

Figure 2 shows a modification in which a single endless belt is employed; and

Figure 3 shows a modified arrangement for cooling the detergent.

Referring now to Figure 1, an endless belt 2 is carried upon and driven by rollers 4 and 6. These rollers are driven (by means not shown) in such fashion as to propel the upper run of the belt 2 from left to right as shown in Figure 1 with any desired velocity. A hopper 8 is provided for the feed solution of the detergent or other composition which is to be processed in accordance with the present invention, and operates to deposit the solution from nozzle 10 communicating there-

with directly onto the upper surface of the moving belt **2**. The detergent solution is spread out in a thin layer or film of the desired thickness on the upper surface of the belt by any suitable means, for example, by a spreading roll **12**, although a knife or doctor blade could also be used for the same purpose.

The moving belt **2** containing the thin film of solution of detergent to be dried then passes through a heating zone which, in Figure 1, takes the form of a heating chamber or section **14** located just below the upper run of the belt **2**. This is heated by any desired means such as by steam introduced via line **16**, with spent steam and condensate removed via line **18**.

Instead of steam chamber **14** or, if desired, in conjunction therewith, a bank of infra-red lamps **20** may be provided above and adjacent the belt in such fashion as to focus heat upon the moving film of detergent passing on the belt just below the lamps. It will be understood that the forward movement of the belt passing through the heating zone, the supply of steam or other heating fluid to the heating section **14** and the number, location and intensity of the infra-red lamps **20** may be so coordinated (either separately or severally) that the solvent initially contained in the feed solution of detergent charged to the process is evaporated to any desired predetermined extent.

In this fashion a substantially dry product is obtained at the end of the upper run of belt **2** and such product may be removed from the belt by means of doctor blade **22**.

The hot dry product thus obtained is then deflected immediately to the upper run of a second moving endless belt **24** which is supported upon and driven by rollers **26** and **28** in exactly the same manner as belt **2**. However, the upper run of this moving belt **24** passes in close proximity to a cooling or refrigeration section **30**, which may take the form of a chamber through which a cooling liquid or refrigerant is introduced via line **32** and removed via line **34**. The refrigeration chamber **30** is in close proximity to the upper run of the endless belt **24** and accordingly heat transfer takes place in such fashion as to reduce the temperature of the hot product derived from the upper moving belt **2** as it is later carried along on the upper run of the lower moving belt **24**.

If desired, the material carried by the upper run of the moving belt **24** may be pressed close to the belt surface, thereby insuring uniformity of thickness and sufficiency of heat exchange, by means of a roller **36** which is so positioned as to bring the outer surface of the roller into the desired relationship with the upper surface of the moving belt **24**.

The cool dried product is removed from the belt surface **24** by means of doctor blade **38** which, if desired, may take the form of a serrated knife which removes the cooled dried detergent product in the form of easily handled ribbons.

Figure 2 shows in schematic form, and partly broken away, a functionally similar arrangement except that only one continuous belt surface is employed, this one belt carrying the feed solution of detergent to be dried successively through a heating zone and then a cooling zone.

In this modification the feed solution of detergent flows from hopper **50** and nozzle **52** onto the upper surface of the continuously moving belt **54**. The belt **54** is carried between and driven by rollers **56** and **58**. The solution of detergent is spread out into a thin film of predetermined thickness by means of a spreading roll **60**. The thin film of material is passed successively through a heating zone and then a cooling zone, the former comprising a heating section **62** into which a suitable heating fluid is introduced via line **64** and withdrawn via line **66** and the latter comprising a cooling section **68** into which a suitable cooling fluid is introduced via line **70** and withdrawn via line **72**. The cooled dried product is removed from the end of the upper run of the continuously moving belt **54** by means of a doctor blade **74** from which it is taken for further processing and/or packaging as desired.

Figure 2 does not illustrate a bank of infra-red drying lamps in the heating zone but it should be understood that if desired such infra-red lamps may be employed instead of, or in addition to, the heating section **62** through which fluid such as steam is passed during the process.

Figure 3 constitutes a detail illustrating schematically an alternative method of cooling in which the hot material carried by belt **24** (of Figure 1) is reduced in temperature by directing a spray of cold water or other cooling liquid on the under surface of the belt by means of a perforated pipe **80** to which cold water or other cooling liquid is introduced under pressure. In view of the high heat conductivity of the metal constituting the belt surface, this represents a very desirable means of cooling.

It will be appreciated that the spray cooling means of Figure 3 may be employed in conjunction with or instead of the cooling chamber arrangement shown specifically in Figures 1 and 2.

In addition to, or instead of, any of the heating or cooling means described above, it will be understood that the desired heating and/or cooling may be effected by enclosing the heating and/or cooling sections of the moving belt surface in separate chambers through which rapid currents of a hot fluid such as hot air and a cooling fluid such as cold air may be circulated, respectively, at rates such as will bring about the desired heating or cooling.

The moving belts, and especially their working surfaces, are preferably made of a suitable corrosion-resistant material, such as stainless steel or Inconel. If desired, under certain circumstances it may be preferred to apply a coating of a still more corrosion-resistant metal such as silver, chromium, tantalum, or the like to the working surfaces of the moving belts. Other corrosion-resistant metals may be employed for the working surfaces.

It will be understood by those skilled in the art that the rate of travel of the continuous belt or belts may be varied within rather wide limits and that the temperatures used for drying may also be varied widely depending upon the boiling point requirements of the solvent used, the melting point of the dried product, and the quantity of material undergoing treatment per unit time. By suitably varying the length and width of the belt or belts as well as the lengths of the heating zones and/or the cooling zones, it is possible to dry and properly collect all types of detergent materials and compositions which have a tendency to melt at the temperature at which drying normally takes place, but which at room temperature are solids. However, the extremely rapid rate of drying which can be obtained in accordance with the process of the present invention, particularly by heating the moving belt surface from both sides and consequently exposing a comparatively thin film to concentrated heat sources from both sides, enables almost instantaneous drying of many products which heretofore could only be dried by spray-drying methods or by vacuum-drying methods at much lower temperatures and with many more operating difficulties.

When alcohol is the sole or principal solvent (other than water) in which the detergent material is initially dissolved or dispersed, a suitable system for solvent recovery such as is well known to those skilled in the art may also be employed. This, for example, may take the form of a substantially gas tight enclosure surrounding the continuous belt system, or at least the heating portion of such system, in order to collect the vapors given off during the drying operation, such vapors then being introduced into a suitable condensing system for the recovery of solvent in liquid form. For brevity, the step of removing solvent from the detergent solution may be sometimes referred to hereinafter and in the claims as "dehydration" irrespective of whether the solvent in any

given instance is water, alcohol, or a mixture of water and alcohol.

Synthetic organic detergent solutions which are particularly amenable to treatment according to the present invention are solutions which contain as at least part of the solvent a lower alcohol, such as methyl, ethyl, and isopropyl alcohols.

While the process of the present invention is applicable to a wide variety of synthetic organic detergent compositions and to other compositions having similar properties, it has been found to be particularly applicable to the drying and cooling of alkali metal salts, such as the sodium salt, of sulphated partial esters of a polyhydroxy alcohol and fatty acids having from 8 to 24 carbon atoms. Sodium salts of sulphated monoglycerides of fatty acids such as those derived from vegetable and animal oils of the type of coconut oil, hydrogenated tallow fatty acids and the like are particularly suitable, especially when they have been preliminarily treated so as to render them substantially inorganic-salt-free.

The process of the present invention is particularly well adapted to dry or dehydrate and collect on a commercial scale synthetic organic detergent compositions (especially inorganic-salt-free) of the kind described herein starting from solutions having an initial solids content of approximately 40%, say from about 20 to 50%, and ending with a substantially solid detergent composition containing about 3% or less by weight of moisture.

While specific examples of preferred methods and apparatus embodying the present invention have been described above, it will be apparent that many changes and modifications may be made therein without departing from the spirit of the invention. It will therefore be understood that the examples recited and the particular apparatus and methods of procedure set forth above are intended to be illustrative only, and are not intended to limit the invention.

What is claimed is:

1. The method of drying a solution of a substantially inorganic-salt-free synthetic detergent composition having as an active ingredient a sulphated organic compound which has a tendency to melt at the temperature at which drying takes place but which is solid at room temperature,

which comprises spreading said solution in the form of a relatively thin layer on the surface of a continuously moving belt, passing said belt containing said layer through a heating zone and heating the solution at least partially by radiant heating from above while carried on said belt sufficiently to reduce the solvent content thereof to not more than about 3%, passing the resulting detergent composition of reduced solvent content through a cooling zone while carried on the surface of a continuously moving belt to effect cooling and solidification thereof, and thereafter collecting the solidified dried detergent composition from the cooling zone.

2. A method as defined in claim 1 in which both the heating and the cooling steps are performed while the material undergoing treatment rests on the surface of the same continuously moving belt.

3. A method as defined in claim 2 in which the initial solution of substantially inorganic-salt-free synthetic detergent composition has a solids content of about 20 to 50%.

References Cited in the file of this patent

UNITED STATES PATENTS

525,572	Andrae	Sept. 4, 1894
916,319	Iveson et al.	Mar. 23, 1909
1,413,786	Robertson	Apr. 25, 1922
1,608,147	Vierling	Nov. 23, 1926
2,131,666	McDougall	Sept. 27, 1938
2,284,848	Ryan	June 2, 1942
2,330,922	Riegler	Oct. 5, 1943
2,360,257	Muller et al.	Oct. 10, 1044
2,419,875	Birdseye	Apr. 29, 1947
2,423,450	Heald et al.	July 8, 1947
2,423,452	Holuba	July 8, 1947
2,488,997	Thornburg	Nov. 22, 1949
2,521,442	Birdseye	Sept. 5, 1950
2,529,704	Olsen	Nov. 14, 1950
2,581,081	De Vout	Jan. 1, 1952

FOREIGN PATENTS

883,178	France	June 25, 1943
475,580	Canada	July 31, 1951