This invention relates to the solidification of liquids by the simultaneous atomizing and cooling of the liquids so that the liquid is entirely or substantially entirely converted into a dry powder or other more or less finely subdivided condition.

The material to be treated may be one which is normally solid at room temperatures, and which is melted or otherwise brought to a liquid condition prior to its treatment in my process. Typical of such materials are natural and synthetic resins and gums, waxes, paraffin, soap, phenol, urea, dichlorobenzene, and higher fatty acids such as stearic acid. The process also is suitable for the solidification of materials normally liquid at room temperatures, such as instance as milk, fruit juices and analogous food products. It is equally suitable for the preparation of any other material in a finely subdivided from such as any of the metals, whether their melting point be high or low. This invention covers the method of separating any desired material into a finely subdivided form by atomizing the material in its liquid state in intimate contact with a suitable second liquid whose boiling point is lower than the melting point of the material being subdivided, which second liquid acts as a refrigerant for the purpose of solidifying the atomized first liquid material. While the invention is applicable to practically all materials which may be melted, in the description of the process a material is selected which is normally solid at room temperature and the refrigerant material is of necessity one which is normally gaseous at room temperature.

In accordance with the invention, a stream or jet of the material in liquid form is atomized and the spray or fine drops in suspension are solidified by the action of a refrigerant gas which is also delivered in liquid form and vaporized to produce the cooling effect and the solidification of the liquid material. The extent of sub-division of the solid product may be controlled by varying the violence or direction of the jet and by the juxtaposition of the jet of the expanding refrigerant. The process is characterized by complete or substantially complete and practically instantaneous solidification of the material to be processed, so that no liquid residue remains in the product which is formed, the complete vaporization or gasification of the liquefied refrigerant, and the complete or substantially complete separation of the product and the gas.

The product may be drawn off continuously or intermittently and the refrigerant gas, typically propane, carbon-dioxide, ammonia, sulphur-dioxide or equivalent material is preferably continuously circulated through a closed system, the cold expanded gas being requalified and reused. The refrigerant selected should preferably be one which does not dissolve in or react with the material being solidified.

The solidification of the type of materials first listed above into powdered or other more or less finely subdivided solid form, by the present process is advantageous for many reasons. Certain of the solids for instance the resins and gums, are but slowly soluble in the solvents with which they are commonly employed. Uniform mechanical sub-division of the materials to facilitate their solution in solvents is both difficult and expensive. By my present invention fine sub-division of these materials is accomplished with expedition, facility and unusual economy because my invention utilizes to the greatest degree the 20 latent heat of vaporization of the refrigerant because of the extremely intimate contact possible in the mixture of two atomized liquids.

In the case of solidification of liquids which contain a large amount of water such for instance as milk or fruit juices, the process is superior to ordinary freezing methods, because the entire product is actually frozen instantly without separation of ingredients of different freezing points, as opposed to freezing merely a mass of ice crystals between which drops or bodies of concentrated liquid are entrapped. Such materials furthermore, even at extremely low temperatures, do not lend themselves to solidification in bulk and subsequent mechanical subdivision.

By using sufficient liquefied refrigerant gas to effect complete solidification of the material being treated, the resultant product will be uniform, homogeneous, dry and powdery and not sticky or with traces of liquid remaining in it,—this to the end that the product may be readily measured, conveyed or packaged without supplemental processing.

The spray of the liquid material to be solidified and the spray of the liquid to be gassified may be relatively formed and positioned in various different ways. Means is preferably provided for filtering out any extremely minute solid particles which might be wafted along with the gas stream as it leaves the processing chamber and in accordance with certain embodiments of the invention.

Separation of the solid product from the gaseous solidifying agent is accomplished gravitationally, and the formation and withdrawal of...
the solid material carried on either continuously or intermittently as desired. The process does not involve the development of substantial pressures in the processing chamber and in fact in most cases a pressure approximating that of the atmosphere may prevail therein.

The invention may be more fully understood from the following description in connection with the accompanying drawing: In this drawing:

Fig. 1 is a diagrammatic vertical sectional view through an apparatus which may be employed in carrying out the invention, and

Figs. 2, 3 and 4 are sectional views showing different forms of nozzle arrangements for spraying the two liquids.

I have illustrated in Fig. 1 a chamber 10 into the upper end of which extends a nozzle 11, the orifice of which is controlled by a needle valve 12. Liquefied refrigerant gas from a reservoir 13 flows under pressure to the nozzle 11 through a pipe 14 and vaporizes in the chamber 10. Arranged closely adjacent to the refrigerant nozzle 11 is a nozzle 15 through which the liquid to be solidified enters the chamber. This liquid may flow to the nozzle 15 through a pipe 16 from a suitable source of supply such as the reservoir 17. I have shown the nozzle 15 provided with a heating means such as a jacket 18 for steam or the like, the jacket being maintained at a temperature which will prevent any premature solidification of the material and the consequent clogging of the nozzle 15. The container 17 and pipe 16 may be similarly jacketed or otherwise heated if necessary where normally solid material is to be kept in hot liquid form prior to its delivery into the chamber. The liquid in the reservoir 17 may be pumped under pressure by a pump 19a through the nozzle 15, or may be forced through by gravity or by the pressure of gas forced into said reservoir through a pipe 19b which may also be used to fill the reservoir.

The chamber is shown with a tapered bottom portion 19c leading to an outlet 10b which may be controlled by an outlet valve 10c. The subdivided solid (which is the product of the process) may be removed continuously or intermittently, as desired, through the outlet 10b, or there may be provided a screw conveyor or other means for removing the product from the chamber.

The vaporized refrigerant passes from the chamber 10 through the conduit 19 to any suitable re-liquefying apparatus. For illustrative purposes I have shown the system as including a compressor 20, a drier 21 which may contain silica gel or the like, and a condenser 22 from which the liquefied refrigerant is returned to the reservoir 13.

In order to prevent minute solid particles from entering the conduit 19, there may be provided a separator 23 located in or at the upper end of the chamber 10, and communicating with the conduit 19 but having no communication with the main chamber 10 save through one or more filters. These are shown as cloth cylinders 24 in the chamber 23. They are closed at their upper ends and open at their lower ends into the chamber 10. The walls of these cylinders are pervious to the gas so that it can flow there-through to the outlet 19, but are impervious to the solids. Solids collecting on the cylinder walls may be conveniently removed as by jogging or otherwise agitating the cloth, mechanism for this purpose being diagrammatically illustrated and including a jogger bar 25 pivoted at 26 on the wall of the casing 10. This bar is connected by links 27 to the solid tops 28 of the cylinders, and the outer ends constitute a projecting operating handle 29.

Figs. 2, 3 and 4 illustrate three of many possible alternative nozzle arrangements which may be utilized for injecting the two liquids into the chamber 10 and assuring the desired sub-division of the liquid material immediately prior to its solidification.

In Fig. 2 the liquefied refrigerant flowing through pipe 30 enters a nozzle 31 controlled by a needle valve 32. The liquid to be solidified passes through a pipe 33 into an annular jacket 34 encircling the nozzle 31 and having an annular inwardly converging outlet 35 arranged closely adjacent to the discharge orifice 36 of the nozzle 31. The annular jacket 34 may be encircled by a heating jacket 37 or other means may be resorted to for preventing premature solidification of the liquid to be solidified. With this arrangement the expanding stream of refrigerant issuing from the orifice 36 is operative to draw, by an injector action in an annular stream of liquid to be solidified, through the opening 38. Thus, the solidification of the liquid to be solidified occurs at the nozzle 35 and intimate commingling of the expanding refrigerant gas and finely divided liquid particles assures instant and complete solidification.

Fig. 3 shows an alternative type of construction which is particularly adapted for use in cases where the refrigerant has a relatively low vapor pressure at the temperature of the liquid to be cooled. In this figure, 38 is a nozzle, with a heating jacket 39 and controlled by a needle valve 40. The liquid refrigerant flowing through pipe 41 is and the liquid to be solidified flowing through pipe 42, are mixed in the nozzle and discharged together therefrom. Valves such as 43 in either or both lines may be employed, the valve shown being in the refrigerant line 41 to regulate the flow of the refrigerant.

Fig. 4 illustrates another alternative type of construction in which the nozzle 44 from which the liquefied refrigerant is discharged at high velocity is closely adjacent to a nozzle 45 through which the material to be liquefied enters the chamber 10. The nozzle may likewise be jacketed and the liquid may be pumped to the nozzle 45 if desired, or the jet from the nozzle 44 may act across the liquid to pull the liquid to be solidified out of nozzle 45 by an injector action.

It will be observed that all forms of the invention involve the intimate mixture of two liquids in such a fashion that the physical state of both of them is changed, one liquid being changed from a liquid to a gas and the other from a liquid to a solid, and that in every case the mixture of the liquids is such that complete solidification of one liquid and complete gasification of the other occurs. It is obvious that by selecting the proper temperature and/or pressure at which chambers 10, 13 and 17 are maintained and by selecting the proper refrigerant material my invention is applicable to the solidifying of any desired material supplied in liquid form.

While, as suggested above, the invention is particularly well adapted for the production of a solid in powder form, the proper arrangement of the nozzles and the proper control of temperatures and the use of supplemental nozzles if required permits the size of the solid particles to be varied.

Various and sundry methods of automatic or manual control may be employed to insure the correct supply of refrigerant and/or material de.
pending upon the type of liquid to be solidified, the temperature thereof, the kind of refrigerant, and the pressure at which the liquids are delivered.

The two liquids should be delivered at such relative rates that the temperature in the chamber is maintained below the solidifying point of the material and above the vaporizing point of the liquid refrigerant at the pressures employed.

This temperature may be varied between these limits by varying the relative rates at which the two liquids are supplied. Any one of the supply valves may be operated by hand or one or more of them may be automatically operated by a thermostat in the chamber to increase or decrease the rate of flow of one while the flow in the other is constant, or to vary the two inversely.

Having thus described my invention, what I claim as new and desire to secure by Letters Patent are:

1. A method of converting a liquid into a solid, which includes the step of introducing the liquid to be solidified and a liquefied refrigerant gas into a chamber in such relative proportions that substantially complete solidification of one liquid and complete gasification of the other is effected.

2. A process of converting a liquid into a solid, which includes the step of spraying the liquid to be solidified and a liquefied refrigerant gas into a chamber at points so juxtaposed that the sprayed liquids are sufficiently intimately commingled and in such relative proportions as to effect complete solidification of one liquid and complete gasification of the other.

3. A process of converting a liquid into a subdivided solid, which includes the step of spraying the liquid to be solidified and a liquefied refrigerant gas into a chamber in such relative quantities that the latent heat of solidification of one spray substantially balances the latent heat of gasification of the other spray, and said sprays separate by gravity as a gas and a powder.

4. A method of subdividing a material, which includes intimately contacting the material in its liquid state, with a liquid which boils at a temperature lower than the solidifying temperature of the material, in such relative quantities that the exchange of heat between the material and the liquid will result in the solidification of substantially all the material and vaporization of substantially all the liquid.

5. A method of forming a finely subdivided solid material, which consists in introducing a stream of liquefied solid and a jet of liquefied refrigerant gas into a chamber, the relative proportions being such that the liquefied solid is atomized and substantially completely solidified by the vaporizing refrigerant liquid.

6. A method of forming a liquid into a powder, which consists in introducing a stream of liquid into a chamber and atomizing and freezing all of the constituent parts of the liquid stream by a jet of liquefied refrigerant gas.

7. A method of forming a dry, finely divided solid material, which consists in spraying into a chamber two materials both in liquid form, one having a boiling point below the melting point of the other, and such materials being in such relative proportions that the interchange of heat in the sprays causes one liquid to substantially completely vaporize and the other liquid to substantially completely solidify while in suspension.

8. A method of forming a dry finely divided solid material, which consists in continuously spraying a liquid into a chamber, continuously delivering into said chamber a liquefied refrigerant at such a rate as to thereby substantially completely freeze the spray, separating the refrigerant gas and the solid by gravity, and separately withdrawing the solid and the gas.

9. A method of producing a powder, which includes the steps of flowing a stream of liquid material into a mixing chamber, and directing an expanding jet of liquefied refrigerant gas against said stream.

10. A method of the character described, which consists in introducing a stream of normally solid material and a stream of normally gaseous refrigerant into a mixing chamber while both materials are in liquid form to effect substantially complete solidification and sub-division of one liquid and gasification and expansion of the other liquid.

11. A method of the character described, which consists in discharging in juxtaposed relationship a normally solid material and a normally gaseous refrigerant while both are in liquid form to effect substantially complete solidification and sub-division of one liquid and gasification and expansion of the other liquid.

12. A method of the character described, which consists in discharging in juxtaposed relationship a normally solid material and a normally gaseous refrigerant while both are in liquid form to effect solidification and sub-division of one liquid and gasification and expansion of the other liquid, the refrigerant being delivered in such quantities as to insure a complete solidification of the other liquid.

13. A method of forming a finely subdivided solid material, which consists in introducing a stream of liquefied solid and a jet of liquefied refrigerant gas into a chamber such that the liquefied solid is atomized and solidified by the vaporizing refrigerant liquid, and heating the inlet through which the normally solid liquid passes into the chamber to prevent clogging of the inlet by pre-mature solidification of the material.

14. A process substantially as described, which consists in maintaining a gas in liquefied condition by pressure and maintaining a solid in liquefied condition by heat and discharging the liquids into a mixing chamber at such rates that one liquid is solidified and sub-divided and the other liquid is gasified and expanded.

15. Means for forming a powder of a material normally liquid at room temperatures, which consists in introducing said liquid into the chamber in intimate contact with an expanding jet of liquefied refrigerant gas.

16. Apparatus for making finely subdivided solids, including a chamber, means for conducting the liquid to be solidified into said chamber, and a substantially closed refrigerating system including said chamber, a nozzle through which liquefied refrigerant is introduced into the chamber, means for withdrawing the refrigerant gas from the chamber and reliquefying it, and means for filtering entrained solid particles from the refrigerant gas as it leaves the chamber.

17. Apparatus for solidifying a liquid, including a chamber, a source of liquefied refrigerant gas, a source of liquid to be treated, means for delivering liquids from said two sources into the chamber at points located in close proximity whereby the liquid material to be treated will be finely subdivided and will be solidified by the...
4. refrigerant, and a gas-pervious, solid-impervious material in said chamber and through which the gas escapes from the chamber.

18. Apparatus for solidifying a liquid, including a chamber, a source of liquefied refrigerant gas, a source of liquid to be treated, means for delivering liquids from said two sources into the chamber at points located in close proximity whereby the liquid material to be treated will be finely subdivided and will be solidified by the refrigerant, a gas-pervious, solid-impervious material in said chamber and through which the gas escapes from the chamber, and means for mechanically displacing solids collecting on said material.

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