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S. E. SHEPPARD ET AL

CAKE OF COMPRESSED GELATIN POWDER AND PROCESS OF MAKING THE SAME

Filed April 28, 1926

FIG. 1.

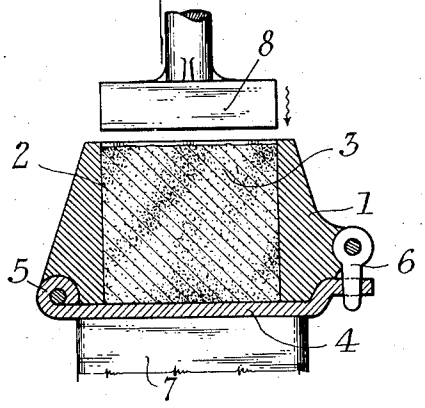


FIG. 2.

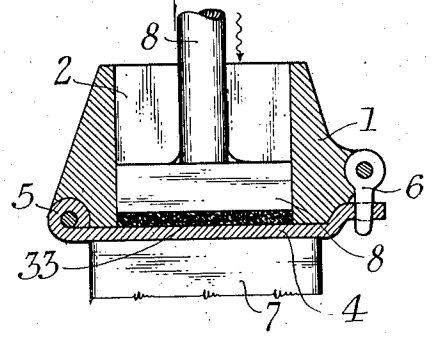
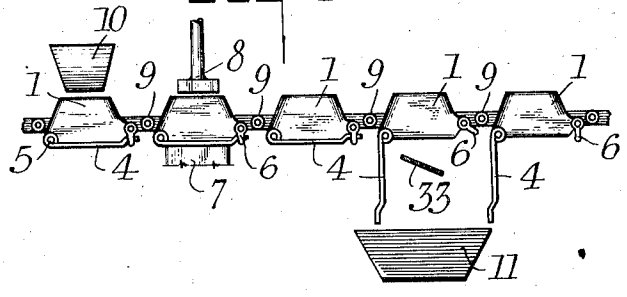


FIG. 3.



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UNITED STATES PATENT OFFICE.

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CAKE OF COMPRESSED GELATIN POWDER AND PROCESS OF MAKING THE SAME.

Application filed April 28, 1926. Serial No. 105,276.

This invention relates to cakes of compressed gelatin powder and to processes of making the same.

One object of the invention is to provide a compressed cake of powdered gelatin in any suitable or desired form. Another object is to provide such a cake in a form which will function without trouble in the processes in which the gelatin is subsequently employed. Another object of the invention is to provide a process for making such cakes which avoids injury by infection with micro-organisms during the process. Still another object of the invention is to provide a process which avoids loss from the sticking of the gelatin to drying supports. Another object of the invention is to provide a process which avoids the expense of chilling operations. A still further object is to provide a process in which the total time of operations is reduced to the minimum. Still another object of the invention is to provide a process for producing the above mentioned cakes without hydrolyzing or otherwise impairing the quantities of the gelatin. Other objects will hereafter appear.

In the accompanying drawing,—

Fig. 1 is a diagrammatic section upon an exaggerated scale showing one form of apparatus in which our process may be carried out, the parts of the apparatus being in initial position.

Fig. 2 is a view similar to Fig. 1 with the parts of the apparatus in the final compression position.

Fig. 3 is a diagrammatic side elevation upon an exaggerated scale of an apparatus in which our process may be carried out continuously.

In the customary manufacture of gelatin, such as for photographic purposes for instance, the source of gelatin, such as hides, are limed, washed, neutralized, cooked, and gelatinous material extracted and clarified, concentrated and bleached. Finally the solution containing the gelatin is chilled and brought into sheet form, these sheets containing large percentages of water. Such sheets are finally dried until the moisture remaining in the gelatin is about 10 to 15%, although this will vary with the season of the year, the specific batch of gelatin, and other factors. The drying is usually carried out with the chilled gelatin sheets located upon nets. The temperature of drying has to be carefully regulated and the process is a rather slow one. If the temperature is raised too high, there is a considerable loss from the sticking of the gelatin to the nets, or even from its melting or running through them. On the other hand, the prolonged drying operation means that very large quantities of air come in contact with the gelatin, so that there is great danger of molds and bacterial growth infecting the product and spoiling it by propagation therein.

We have found that the disadvantages of the older processes such as infection with microorganisms, sticking to drying nets, the expense of chilling operations, and undue length of time of the process, can be overcome by spray drying the gelatin to produce very quickly a fine powder of the proper water content, and then compressing this powder into cakes which may have any suitable form. In the preferred embodiment of our invention, these cakes are in the form of sheets and the compression is such that their specific gravity is greater than that of water. In other words, they will sink in water.

We have also found that the result is not satisfactory if the gelatin be merely spray-dried and left in the form of a fine powder. The latter is very voluminous, being approximately ten times the volume of an equal weight of leaf gelatin. Aside from the excessive storage space required, this gelatin is surprisingly difficult to dissolve. When, for instance, it is placed in water agitated by a mechanical stirrer, it floats on the surface and forms large sized clumps which become wet on the outside so as to form a skin with a dry powdered core, the voids of said core being full of air. These bothersome floating clumps require a far longer time before they are thoroughly wetted and the air in them displaced, than it would take to completely dissolve ordinary leaf gelatin. In other words, the advantages of spray drying would be largely lost if the product remained in this unsatisfactory state.

We have found that compressing of this powder under the proper conditions will produce cakes of any desired shape which will dissolve in water with at least the

rapidity of ordinary leaf or sheet gelatin of commerce, and yet can be produced by a process which overcomes the difficulties in the usual process, hereinabove described. While we shall describe certain forms of our invention by way of illustration, it will be understood that this invention is not restricted to the details, thus given, except as indicated in the appended claims.

In the first operation of our process, we spray dry into air an aqueous solution of gelatin which has been previously obtained in proper condition from the hides or other sources by the usual expedients. The concentration of the gelatin in the water just prior to spraying may vary over a considerable range, 5 or 6% solutions being illustrative. Any suitable spray drying apparatus may be employed, typical ones being illustrated for instance in U. S. Letters Patent No. 1,183,098, Merrell et al., May 16, 1916, desiccating apparatus, No. 1,141,879, Merrell et al., June 1, 1915, desiccating process, and No. 1,183,393, Merrell et al., May 16, 1916, spraying nozzle, these patents applying the general principle of 666,711, Stauf, Jan. 29, 1901, method of desiccating blood.

The liquid solution of gelatin is forced, under pressure, through a nozzle into a spray which is affected by currents of air, so that it spreads out into a dry chamber where the air takes up moisture, leaving a very voluminous powder, the very fine particles of which contain approximately 10% of moisture or even as low as 5%. Of course, other gaseous vehicles for carrying away the moisture can be employed instead of air, and the temperature of the air is kept below the point at which the quality of the gelatin would be impaired by hydrolysis or other harmful heat effects. While the process can be carried out with the air in the drying chamber at atmospheric pressure, lower working temperatures can be employed by maintaining a partial vacuum in the drying chamber.

In the next step of our process, the spray dried gelatin is compressed into cakes of any suitable forms and dimensions. It may, for example, be placed in metal forms or boxes and compressed by a plunger operating thereon under hydraulic pressure. For instance, when operating upon a spray dried powder which is so light that it has a volume about 10 times greater than that of leaf gelatin, we can obtain firm cakes which can be packaged readily by subjecting the powder to a pressure of 1200 lbs. to the square inch. Cakes or sheets made under such a pressure have a specific gravity which is less than that of water and will consequently float. Since it is desirable to facilitate the dissolving of the cakes in water when the former are used, say in photographic work, we increase the density of the cakes, so that their specific gravity is greater than that of water, thus causing them to sink therein. In the preferred form of our invention, we submit the above described powder to a pressure of roughly 50,000 to 60,000 pounds per square inch. The cakes or sheets thus obtained not only sink in water readily, but dissolve in as short a time as leaf gelatin.

The percentage of water in the powder when it is compressed has a considerable effect upon the pressure which is required. The pressures noted above have been employed satisfactorily where the powdered gelatin contained approximately 12% of moisture. By increasing the moisture of the gelatin, for example to 15% or even 20%, the necessary pressures for obtaining compact sinking cakes or sheets can be greatly lowered.

In the simplest and yet effective form of our invention, the spray dried powder is compressed without any intermediate operation. We have found it useful to interpose a simple and inexpensive treatment which facilitates the preparation of the cakes. We wet down the powder with an approximately non-aqueous, non-solvent liquid, which is preferably miscible with water, such as ethyl alcohol, for example. Such alcohol may be of the usual concentrated commercial type. The excess of the liquid can be drained away before compression, or it can be squeezed out during the compression step. When the powder has been treated with such a liquid, the pressure required to produce coherent cakes, having specific gravity greater than water, is much lower than the figures hereinabove given by way of example.

We greatly prefer a spray drying operation for our process because it not only avoids the difficulties of the ordinary process, as explained above, but it provides the gelatin powder in an especially pure form. If, for example, we should attempt to produce the powder by grinding up ordinary gelatin leaves, there would be great danger of the powder being contaminated by metallic particles abraded from the grinding surfaces. It is particularly important, in the case of photographic gelatin, that the latter be entirely free from metallic particles, such as iron for example.

Our process may be carried out by a great many different kinds of apparatus. That shown in the drawing is merely one useful form. Referring to Fig. 1, a strong metal form or box 1 is provided with a rectangular opening 2 in which is located the voluminous powdered spray dried gelatin 3. The box is provided with a bottom 4 which is hinged at 5 and provided with any suitable or preferred locking device 6. A lock that slightly cams the bottom 4 into position is

the best. The box 1 is located upon the lower member 7 of a hydraulic press. Coacting with it is the other member of the press 8.

With the box full of powder, the hydraulic pressure is applied to bring the parts 7 and 8 toward each other with the necessary pressure. The voluminous powder 3 is thereby compressed into a very compact, coherent cake 33, shown in Fig. 2. This cake may be conveniently removed from the box by withdrawing the member 8 from it, removing the box from member 7 and opening the bottom by releasing latch or lock 6 and allowing the bottom to swing downwardly about hinge 5.

As shown in Fig. 3, this process may be carried out in a practically continuous manner by arranging the boxes 1 in the form of an endless chain, a portion of which is indicated in Fig. 3, suitable linkages between the boxes being indicated at 9. In this way the powder may be loaded in at one station from a hopper 10, the powder compressed by means of members 7 and 8 at another station and then completed cake 33 discharged from the box 1 into a suitable container 11 at a still different station. Thus the boxes will be carried through a regular cycle.

Having thus described our invention, what we claim as new and desire to secure by Letters Patent is:

1. The process of preparing coherent gelatin cakes which comprises spray drying gelatin to form a voluminous powder and compressing such powder into the form of said cakes.

2. The process of preparing coherent gelatin cakes which comprises simultaneously comminuting an aqueous gelatin solution and drying the comminuted particles thereof to a voluminous powder and compressing this powder into the form of said cakes until the specific gravity of the compressed powder is at least as high as that of water.

3. The process of producing coherent gelatin cakes which comprises spray drying said gelatin to produce a voluminous powder containing from 5 to 20% of moisture, and subjecting said powder to a pressure from 1200 to 60,000 pounds per square inch, until said cakes are produced.

4. The process of preparing coherent gelatin cakes which comprises spray drying said gelatin to produce a voluminous powder having a specific gravity less than that of water, and compressing said powder into said cakes, the density of the cakes being sufficient to cause them to sink when immersed in water.

5. The process of preparing coherent gelatin cakes which comprises spray drying said gelatin to produce a powder, the volume of which is of the order of 10 times the volume of an equal weight of leaf gelatin, and subjecting said powder to a pressure of from 1200 to 60,000 pounds per square inch to reduce the volume of the powder and cohere its particles into said cakes.

6. The process of producing coherent gelatin cakes which comprises spray drying said gelatin to produce a voluminous powder, wetting said powder with an approximately non-aqueous, non-solvent liquid miscible with water, and compressing said powder into said cakes, the excess of said liquid being removed prior to the completion of the cakes.

7. The process of producing coherent gelatin cakes which comprises spray drying said gelatin to form a voluminous powder containing from 5 to 20% of water, wetting said powder with ethyl alcohol, and compressing said powder into cakes under sufficient pressure to give the cakes a specific gravity at least as high as that of water, the excess of said alcohol being removed, prior to the completion of the cakes.

8. As an article of manufacture, a coherent cake of gelatin powder, the particles of which are compressed to adhere together, said particles containing from 5 to 20% of water.

9. As an article of manufacture, a coherent cake of gelatin powder particles, compressed to adhere together, said cake having a specific gravity greater than that of water.

Signed at Rochester, New York, this 24th day of April, 1926.

SAMUEL E. SHEPPARD.
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