

Oct. 14, 1947.

R. W. GUNNELL

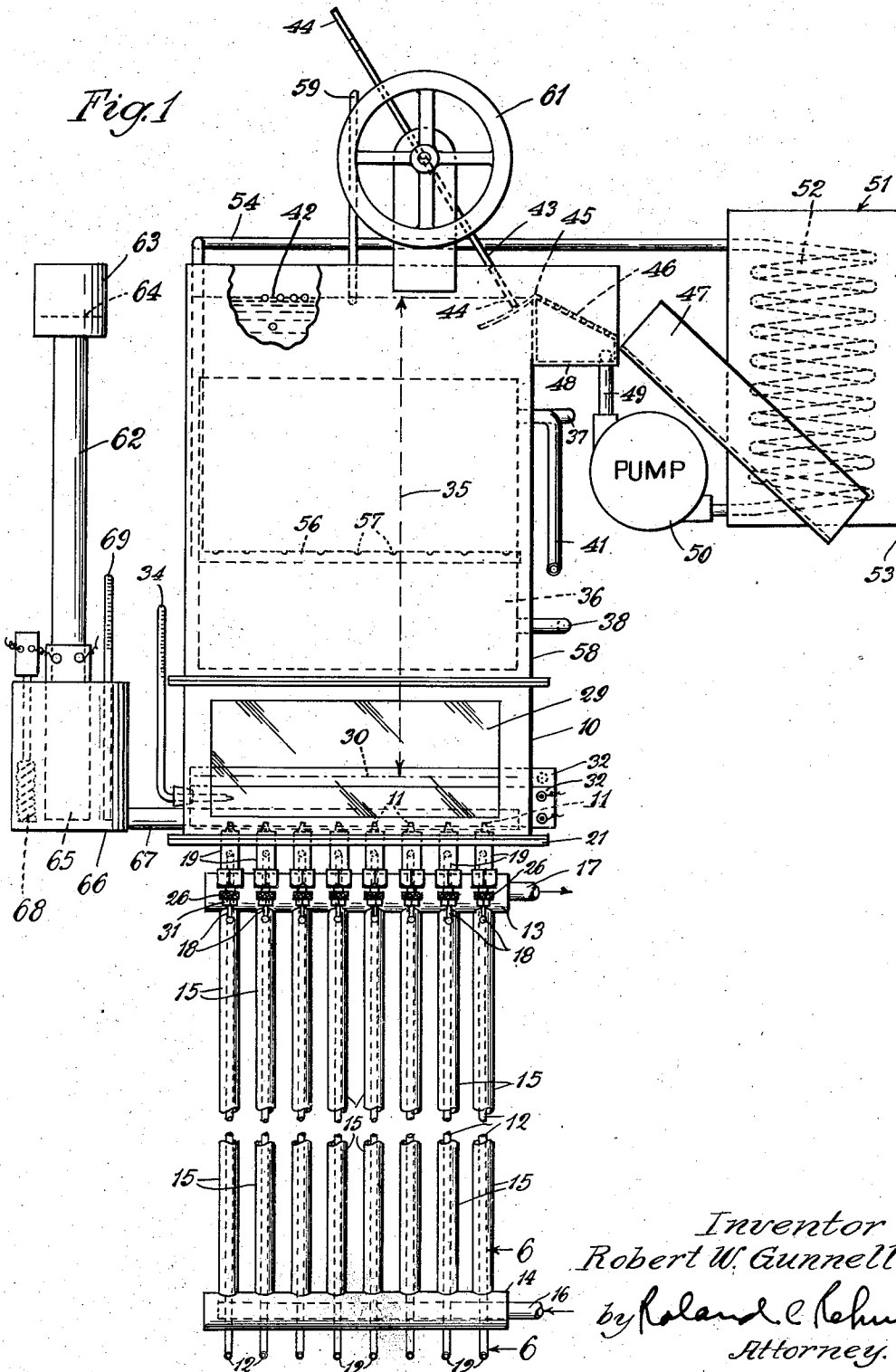
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METHOD AND APPARATUS FOR PRODUCING CAPSULES

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4 Sheets-Sheet 1

Fig. 1



Inventor
Robert W. Gunnell
by Roland C. Behm
Attorney.

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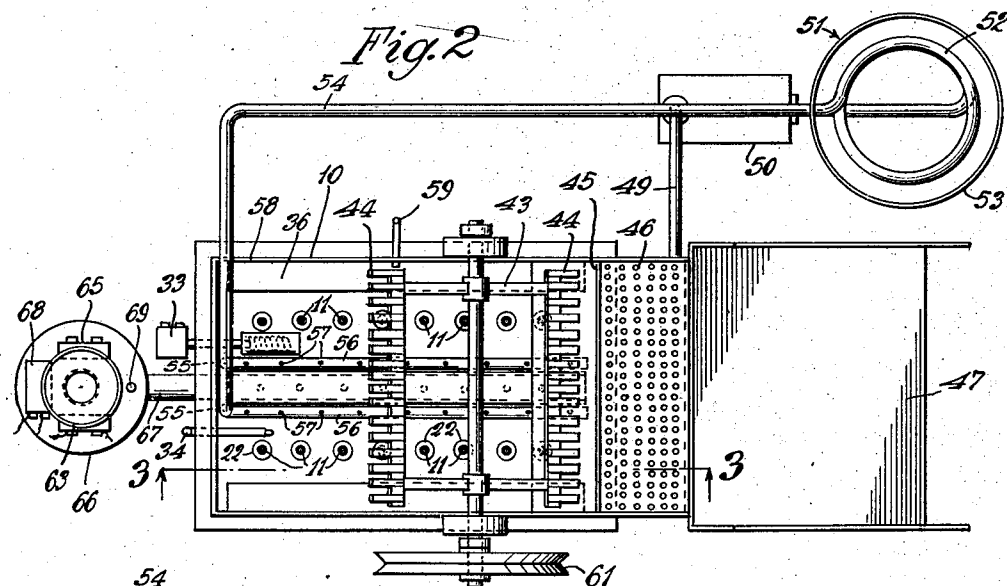
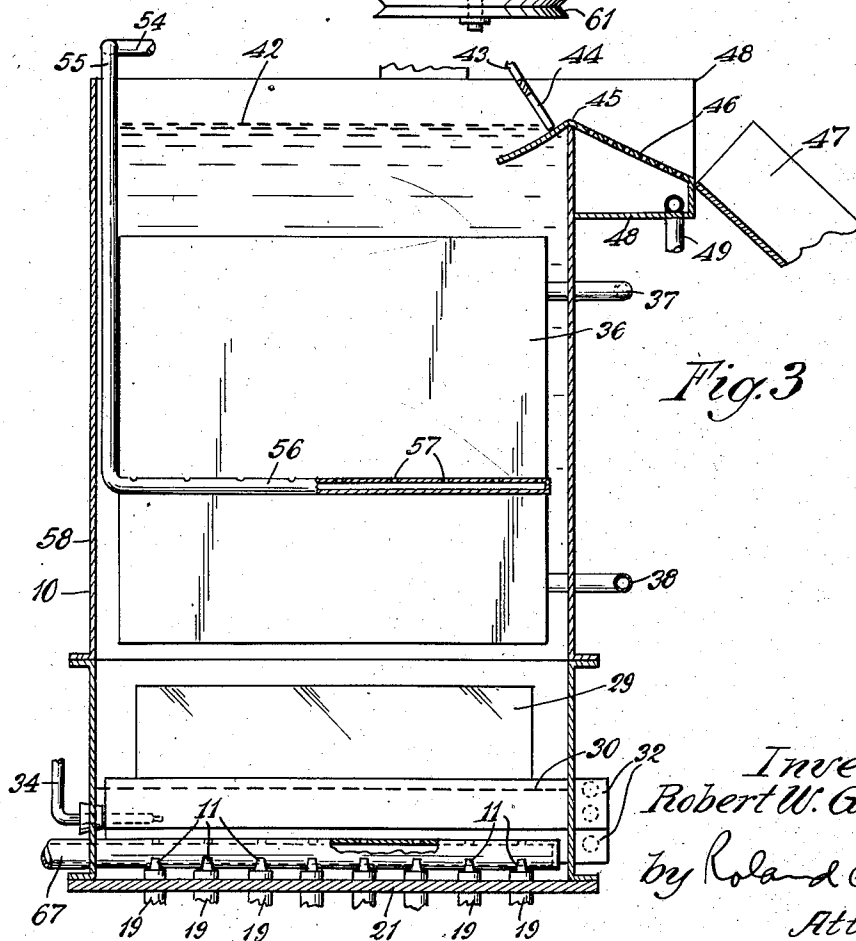
R. W. GUNNELL

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4 Sheets-Sheet 2



Inventor
Robert W. Gunnell
by Roland E. Behn
Attorney.

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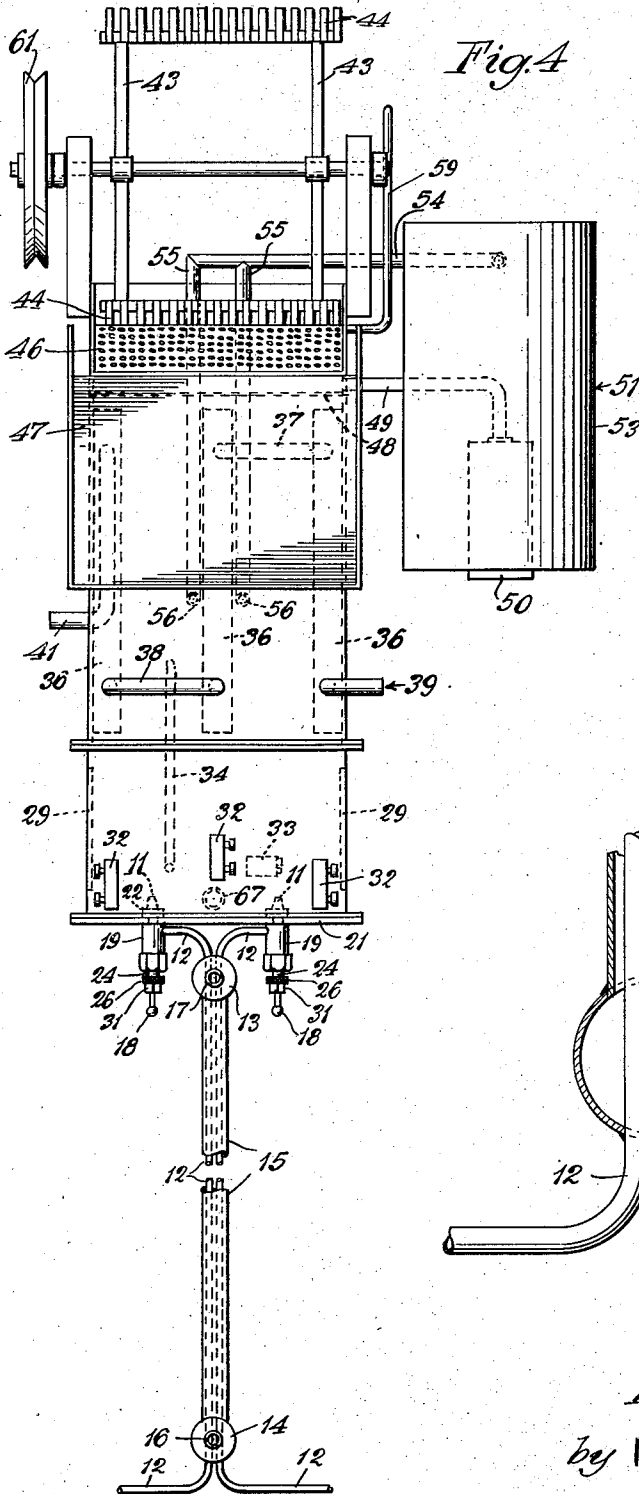
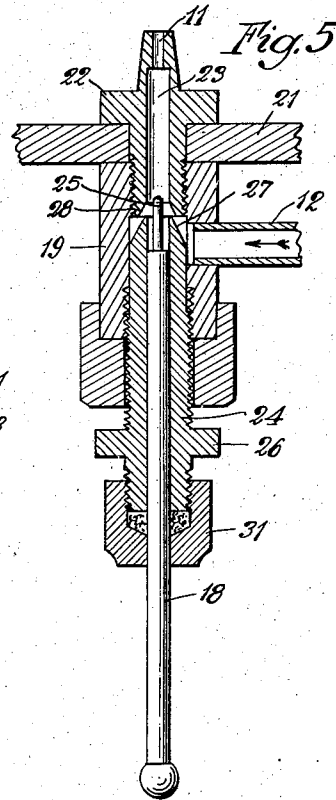
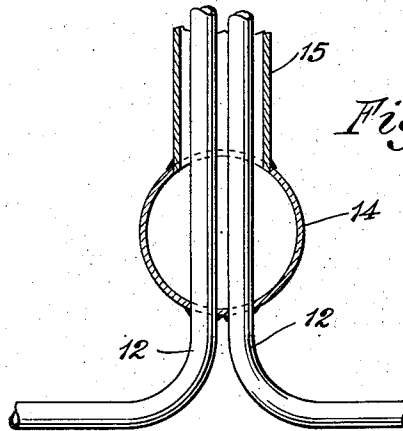
R. W. GUNNELL

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Inventor
Robert W. Gunnell
by Roland E. Behn
Attorney.

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R. W. GUNNELL

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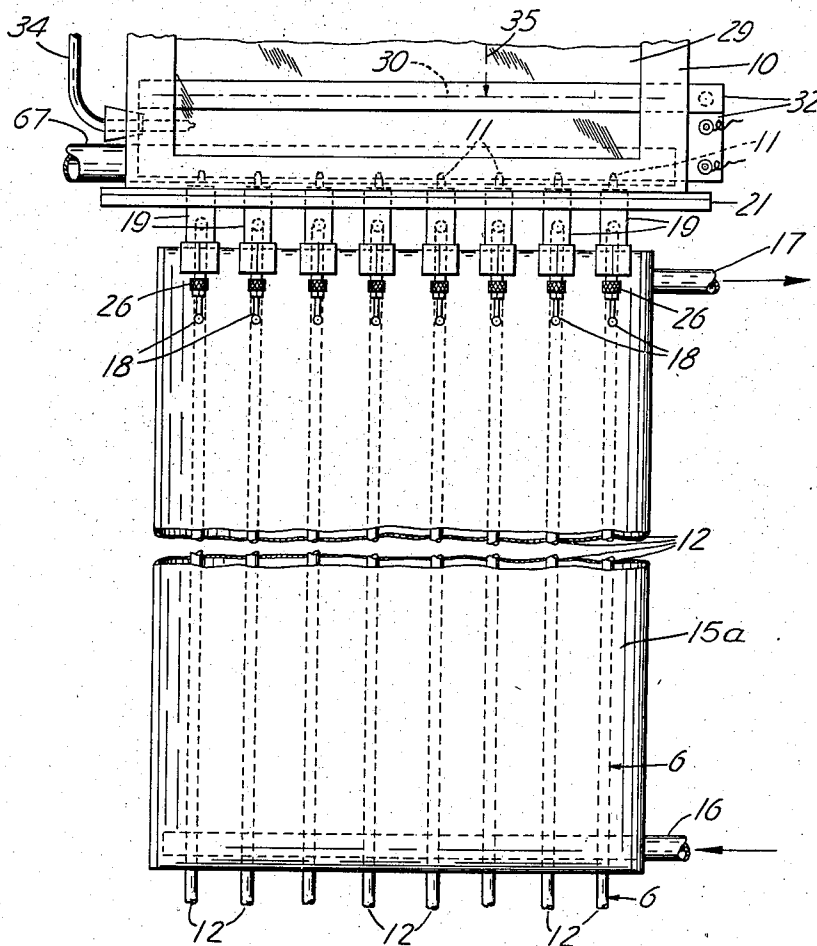


Fig. 7

Robert W. Gunnell
INVENTOR

BY *Charles W. Prater*
attorney

UNITED STATES PATENT OFFICE

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METHOD AND APPARATUS FOR PRODUCING CAPSULES

Robert W. Gunnell, Detroit, Mich., assignor to Industrial Sound Systems, Inc., Cleveland, Ohio, a corporation of Ohio

Application October 19, 1946, Serial No. 704,476

17 Claims. (Cl. 18—5)

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This invention relates to the manufacture of capsules, and among other objects, aims to provide an improved apparatus and method for producing capsules containing various content substances such as medicaments.

The nature of the invention may be readily understood by reference to one apparatus embodying the invention and shown in the accompanying drawings.

In said drawings:

Figure 1 is a front elevation of the apparatus;

Figure 2 is a plan view thereof;

Figure 3 is a side sectional elevation of the upper portion of the apparatus taken on the plane 3—3 of Figure 2;

Figure 4 is a side elevation of the apparatus viewed from the left hand side of the apparatus as shown in Figure 1;

Figure 5 is a section on an enlarged scale of a discharge jet and associated elements;

Figure 6 is a detail section through a header taken on the plane 6—6 of Figure 1; and

Figure 7 is a fragmentary side elevation of an alternate form of the apparatus embodying a single cooling jacket common to the several tubes which supply content material to the nozzles or orifices.

According to the illustrative apparatus, the content substances to be capsulated are formed into globules or pellets, preferably of measured or predetermined volume and covered with a capsule-forming film or shell such as gelatin. The content substance may advantageously be in liquid form in which case they may be released in liquid globules or drops which, due to their inherent surface tension, form into spherical shape. On the globules or pellets a capsule or coating is formed by solidifying or otherwise setting thereon a retaining film or shell of coating substance. The expression "drops" is used in a general sense and not to define a specific volume of content substance. In the illustrative apparatus, the globules or pellets are introduced into a coating liquid (such as gelatin sol), that is, a liquid containing gelatin (comprising the capsule-forming material) and collect thereon a shell or coating which constitutes the capsule. The globules or pellets are caused to travel through the coating liquid, preferably automatically, and for that purpose the latter is adjusted to have a different gravity from that of the globules or pellets. In the present case, the coating liquid has a specific gravity greater than that of the content substance so that the globules of the latter will rise in the coating liquid. For capsulating with a

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thermoplastic coating material, the content substance is preferably chilled before being introduced into the coating liquid and the latter is maintained at such temperature that a coating of the liquid will set or congeal on the globules as they pass, in this case rise, through the liquid. The temperature differences between that of the globules and the coating liquid depends on the amount of cooling necessary to congeal an adequate film of coating on the globules.

The coated globules are removed from the capsulating material before the latter has an opportunity to affect (i. e., melt in the case of gelatin) or otherwise impair the coating. The resultant capsules are seamless and therefore cannot develop leaks as in the seamed capsules heretofore on the market. In the present case, the capsules are separated from the coating liquid and from each other by passing them through an adjacent body of liquid immiscible with the gelatin sol and which advantageously conditions or treats the capsules in this case by further hardening the coating or shell, so that the capsules will not cohere and may be readily handled.

In the illustrative apparatus for performing the aforesaid operations, the content substance in liquid form is introduced into a chamber 10 containing the capsulating substance in liquid form through a plurality of discharge nozzles, orifices or jets 11 located at the bottom of chamber 10. It will be noted that the nozzles or orifices 11 project into the chamber 10 and the discharge ends thereof preferably project into the layer of coating liquid above the bottom of such layer. As here shown (Figure 4), the discharge orifices 11 are arranged for convenience in a pair of rows. Each orifice is advantageously connected to a separate supply line 12 which leads from a metering pump or supply means (not shown) for delivering or forcing the content substance through the tubes at such predetermined rate as to cause globules of the content substance to form in the discharge orifices 11 of the proper size and at the desired rate. Any appropriate metering pump or other measuring device may be employed for this purpose. Solid or semi-solid pellets of content material are of course introduced into the coating liquid much more simply. As will presently appear, it is preferable if these also rise so that they will leave the coating liquid at its upper surface.

In this case, the content substance is conditioned to the proper temperature before issuing from the orifices by passing the same through a cooling device here shown in the form of upper

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and lower headers 13 and 14 connected by tubes or jackets 15 which surround pairs of the rising tubes 12. A fluid refrigerating or cooling medium is introduced into the lower header through line 16 and leaves through line 17. As shown in Figure 7, a single jacket 15a common to all of the tubes 12 may be employed instead of individual jackets for pairs of rising tubes 12 and by the use of a common jacket, the temperature of the content material passing through the tube 16 may be more efficiently controlled. Whether the jacket 15 or common jacket 15a is employed, it is desirable that the refrigerating jacket extend into close proximity to the orifices or nozzles 11 so that the content material discharged from the orifices will be accurately maintained at the desired temperature.

For coating with gelatin, the pellets or globules, as the case may be, are advantageously cooled to a temperature of 10° to 20° F. The gelatin solution is maintained in a thoroughly fluid condition by holding it at temperatures from 120° to 150° F. and this assures the formation of an adequately thick shell on the globules. It will be understood that the temperature of the gelatin or other coating material used, depends on the thermal characteristic of the coating liquid. Various types or grades of gelatin sols or other coating liquids require different temperature conditions, depending upon the melting or setting point of the coating used.

The orifices 11 are advantageously provided with closing means in the form of slidable pins 18 to fill and close the orifice when the apparatus is not in use to prevent gelatin from congealing in or otherwise clogging the orifice. An appropriate closing device is illustrated in Fig. 5. As there shown, the tube 12 after issuing from the upper header 13 enters a fitting 19 fastened to the bottom 21 of the chamber 10 by means of a shouldered nozzle tip 22 screwed inside the fitting and clamping the intervening bottom to form a leak tight joint. The passage 23 connects the tube 12 with the orifice 11. Also threaded into the lower end of fitting 19 is a regulating valve 24 in alignment with the passage 23 surrounding pin 18 whose extremity 25 is adapted to move into and fill the passage 23 and orifice 11 when the apparatus is not in use. A knurled enlargement 26 on the valve 24 facilitates rotation of the latter to adjust its extremity 27 relative to the seat 28 to supplement the regulation of the flow of content substance and to insure discharge of globules of equal size from all of the orifices 11. Such regulation is simplified by incorporating sight windows 29 in opposite sides of the chamber to expose the discharge of the globules from the orifices for inspection. A packing nut 31 covers the extremity of valve 24 to prevent leakage around the pin 18.

The gelatin sol or other coating liquid is maintained at the proper temperature by heating means in the form of a series of electric heating strips 32 which lie parallel to the rows of orifices. The heating elements are controlled by an appropriate thermostat or regulator 33 located inside the chamber 10 and responsive to the temperature of the coating liquid. A thermometer 34 also advantageously indicates the temperature of the coating liquid.

After passing through and thereby receiving a coating from the coating liquid, the globules are separated from the latter and are advantageously further cooled and otherwise treated to harden or set the coating sufficiently for handling and to

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prevent cohesion. In the present case, they rise through a supernatant body 35 of conditioning or separating liquid which floats on the body of coating liquid. The separating liquid is lighter than the coating liquid and of such character as to be immiscible therewith. For convenience the conditioning and separating liquid has been termed a cooling liquid in the claims, but it should be understood that its function is not always or solely one of cooling.

For a gelatin sol, a separating liquid comprising a light hydrocarbon such as kerosene, naphtha, petroleum ether or the like, mixed with carbon tetrachloride or a chlorinated hydrocarbon such as perchlorethylene, is employed. The mixture is made in such proportions of the light and heavier components as will have any desired specific gravity, ranging from about 1.025 to about 1.055 at 60° F. For other coatings the separating liquid should be such as not to impair the coating.

The preferred specific gravity is such as to insure floating of the conditioning liquid upon the coating liquid employed and yet provide the buoyancy required to cause the capsules to rise therein.

The coating liquid may range in specific gravity from about 1.070 to about 1.100 at 130° F., depending upon its composition and the concentration of gelatin or other base. For example, when the coating liquid employed is a gelatin sol of 1.080 to 1.090 specific gravity at 130° F. the separating liquid preferably is of 1.020 to 1.050 specific gravity at 60° F.

One illustrating gelatin coating sol comprises:

	Per cent by weight
Gelatin (150 to 250 Bloom)-----	21
Glycerine-----	7
Water-----	Balance

The glycerin is employed to reduce the brittleness of the gelatin in the capsule. It is added in the amount of about thirty to forty percent of the weight of the gelatin.

A harder capsule material may be obtained by increasing the gelatin in the above sol to twenty-four percent and the glycerin correspondingly, i. e., to 9.6%.

If the content substance be heavier than the coating liquid, the former may be coated by downward travel through the liquid. In that event it will be desirable to use a cooling liquid at higher gravity than the coating liquid so that the latter will float on the former. If a buffer or insulating layer be used, it should, of course, be lighter than the lower liquid and heavier than the upper, and immiscible with both. Completed capsules may be removed from the bottom of the receptacle by hand, by suction or by a mechanical conveyor.

For conditioning the capsules, the separating liquid is such as to cool or set the coating on the capsules adequately as the latter rises through it. In the case of a gelatin sol coating liquid, temperatures of from 40° to 60° F. for the separating liquid are adequate. While the average temperature of the conditioning liquid is lower than that of the sol, the heat of the latter supplied from the heater elements 32 is sufficient to maintain the temperature at the inter-face between gelatin and conditioning liquid sufficiently high to prevent congealing of the gelatin at the inter-face by the conditioning liquid. For this purpose, heater elements 32 advantageously extend through the inter-faces 30 between the liquids.

Where it is desired to obtain specially thick or hard capsules by the use of a solution of high gelatin or other base concentration, and correspondingly high melting or setting point, congealing at the inter-face which might otherwise occur, is prevented by interposing a layer of an intermediate or buffer liquid between the coating solution and the conditioning liquid and immiscible with both. Such a layer of intermediate solution of specific gravity lower than that of the coating liquid and higher than that of the conditioning liquid, floats upon the surface of the coating solution and protects or insulates it from direct contact with the cold convection currents descending in the supernatant conditioning liquid. The presence of an intermediate liquid layer also permits the use of a conditioning liquid which would be miscible with the coating liquid if they were in contact.

In such case the intermediate layer may have the composition above described for the conditioning liquid, and the uppermost liquid may consist of water alone or water to which alcohol, acetone, or other soluble substance which will not attack or affect the capsules, may be added to control and regulate the specific gravity within the range of about .985 to about 1.010 at 60° F. At the low temperatures of the conditioning liquid, water does not affect the gelatin of the capsules.

Free passage of the ascending capsules through the interfaces between the various solutions described above is facilitated by the addition to such solutions of small amounts of those substances which reduce inter-facial tension. These substances may be ordinary soaps or any of the well-known "wetting agents" such as the sulphonated derivatives of the higher alcohols, salts of the sulphonated alkyl aryl compounds and the like. They are added to any or each of the above solutions in amounts preferably ranging from about .017 to about .25% of the weight of the solution, depending upon the practical effectiveness of the particular wetting agent employed.

The conditioning liquid is maintained at the proper temperature by cooling cells 36 in the form of hollow plates, which in this case lie parallel to and on opposite sides of the paths traveled by the rising capsules. The cells are connected in series by the conduits 37 and 38. A liquid refrigerant or a cooling medium enters the first cell through the pipe 39 and leaves the last cell through the pipe 41. Appropriate means (not shown) for cooling and circulating the refrigerant through the cells are provided.

When the capsules reach the surface 42 of the conditioning liquid, they are removed, in this case, by a discharging device in the form of a rotating paddle wheel 43 carrying a plurality of fingers 44 which rake the globules to the overflow weir 45 and cause them to roll down a perforated draining plate 46 into a collecting receptacle 47. The conditioning liquid adhering to the capsules drains through the plate into the collecting chamber 48 as also does the liquid which flows over weir 45. From this chamber, the liquid is withdrawn through line 49 by pump 50 and recirculated, first passing through a cooling unit 51 in the form of a coil 52 immersed in a vessel 53 containing a cooling or refrigerating medium. From the refrigerating unit the cooled conditioning liquid is carried through pipe 54 and discharged through branch pipes 55 whose extremities 56 lie on opposite sides of the center cooling cell 36 and are provided with upwardly opening

orifices 57 which induce an upwardly traveling circulation of the conditioning liquid in the chamber 58. A thermometer 59 advantageously indicates the temperature of the conditioning liquid at its surface.

The paddle wheel 43 is rotated in this instance by a pulley 61 to which an appropriate driving means, not shown, is connected.

The hydrostatic head of the conditioning liquid is balanced in the present instance by a stand pipe 62 connected with the coating liquid supply. Since the conditioning liquid has a lower gravity than the coating liquid, the level of coating liquid in the stand pipe is somewhat below the level 42 of the conditioning liquid. Additional coating liquid is introduced in the upper end of the stand pipe 62. For that purpose, a supply chamber 63 is provided wherein a substantially constant level 64 of coating liquid is maintained.

The coating liquid is elevated to the proper temperature by an electric heating element 65 inside the receptacle 66 which is closely adjacent to the chamber 10 and connected thereto by pipe 67. The stand pipe 62 enters the upper end of chamber 66. A thermostat or other thermo-regulator 68 inside the chamber 66 controls the heater elements 65 to maintain a predetermined constant temperature for the supply of coating liquid. This temperature is advantageously indicated by thermometer 69 within chamber 66. Preheating of the coating liquid as aforesaid reduces the load on the heater elements 32 in chamber 10 and thereby makes closer temperature regulation possible.

The foregoing method assures the formation of a coating or shell of uniform thickness regardless of the relative gravities of coating liquid and content substance. The coating is formed by congealing the coating liquid on the content substance progressively. The coating substance does not become a coating until it has congealed in which condition it cannot shift, and additional coating does not build up until the underlying coating has congealed. Since the globules of content substance are of uniform temperature throughout, they abstract heat from the adjacent coating liquid uniformly through the surface and therefore a coating of uniform thickness is built up on the globules.

This application is a continuation in part of my application Serial No. 522,716, filed February 17, 1944, and entitled "Manufacture of capsules."

Obviously, the invention is not limited to the details of the illustrative apparatus since these may be variously modified. Moreover, it is not indispensable that all features of the invention be used conjointly since various features may be used to advantage in different combinations and sub-combinations.

What I claim is:

1. The method of fabricating capsules which comprises introducing into a capsule forming liquid the content substance at temperatures below the congealing point of the capsule forming liquid so that the content substance will congeal thereon a coating of congealed capsule forming material, causing the coated content material to pass out of the coating liquid into a body of cooling liquid whose temperature is below that of the coating liquid and insulating the coating liquid from the cooling liquid by a liquid layer immiscible with both coating and cooling liquids.

2. In capsule forming apparatus, the combination comprising a chamber having therein superposed layers of capsule forming liquid and cooling liquid of different gravity and immiscible with each other, means for introducing content substance into the coating liquid and causing it to pass in succession through the coating and cooling liquids, said coating liquid having therein a wetting agent to reduce the surface tension at the inter-face of the coating and cooling liquids.

3. In capsule forming apparatus, the combination comprising a chamber having therein superposed layers of capsule forming liquid and cooling liquid of different gravity and immiscible with each other, said cooling liquid being lighter than the coating liquid so that it floats thereon, means for introducing content material into the coating liquid and causing it to pass through the coating liquid and then through the cooling liquid, said coating liquid containing a surface active agent to reduce the surface tension at the inter-face of the coating and cooling liquids.

4. Apparatus for fabricating capsules comprising in combination a chamber having therein superposed layers of coating and cooling liquids, means for maintaining the temperature of the cooling liquid lower than that of the coating liquid, and a buffer layer of liquid between the coating and cooling liquids and immiscible with both and of a gravity intermediate that of the cooling and coating liquids, and means for introducing content substance into the coating liquid and causing it to pass through the coating liquid and into the cooling liquid.

5. Apparatus for fabricating capsules comprising in combination a chamber having therein superposed layers of coating and cooling liquids, means for maintaining the temperature of the cooling liquid lower than that of the coating liquid, and a buffer layer of liquid between the coating and cooling liquids and immiscible with both and of a gravity intermediate that of the cooling and coating liquids, said coating liquid having therein a surface active agent to reduce the surface tension at the inter-face of the coating and buffer liquids, and means for introducing content substance into the coating liquid and causing it to pass through the coating liquid and into the cooling liquid.

6. Capsule fabricating apparatus comprising in combination a vessel containing liquid capsule forming material, a series of supply lines connected with said vessel and leading directly to the capsule forming material for introducing the content substance into the capsule forming material, and a refrigerating jacket around said supply lines to cool the content material to a temperature below the congealing temperature of the coating liquid, said refrigerating jacket extending into close proximity to the point where the supply lines lead to the capsule forming material.

7. In capsule fabricating apparatus, the combination comprising a vessel containing congealable capsule forming liquid, said vessel having therein an orifice through which the content material is introduced into the capsule forming liquid, means for introducing content substance through said orifice at temperatures below the congealing point of said coating liquid, and means for plugging said orifices while the apparatus is idle to prevent congealing of capsule forming liquid in said orifice.

8. In apparatus for fabricating capsules, the

combination comprising a vessel having congealable liquid coating material therein, said vessel having a row of orifices through which content substance may be introduced into said coating material, means for cooling the content substance to temperatures below the congealing point of said coating liquid, and heater units in said coating liquid on opposite sides of said row of orifices to maintain the coating material in liquid condition.

9. Capsule fabricating apparatus comprising in combination a vessel containing superposed layers of coating and cooling liquids, the cooling liquid being of lower gravity than said coating liquid, means for maintaining the cooling liquid at a temperature below the congealing point of the coating liquid, and a layer of liquid between said coating liquid and cooling liquid and immiscible with both and of a gravity intermediate that of the coating and cooling liquids for insulating said coating liquid from the cooling liquid to prevent congealing of the coating liquid by the cooling liquid.

10. Capsule fabricating apparatus comprising in combination a vessel containing superposed layers of coating and cooling liquids, the cooling liquid being of lower gravity than said coating liquid, means for maintaining the cooling liquid at a temperature below the congealing point of the coating liquid, means for heating said coating liquid to maintain the same in liquid condition, and a layer of liquid between said coating liquid and cooling liquid and immiscible with both and of a gravity intermediate that of the coating and cooling liquids for insulating said coating liquid from the cooling liquid to prevent congealing of the coating liquid by the cooling liquid, said coating liquid containing a surface active agent for reducing the surface tension at the inter-face of the coating and the insulating layer.

11. In apparatus for fabricating capsules, the combination comprising a vessel, means for introducing congealable capsule forming material in liquid condition to said vessel, means for introducing into said capsule forming liquid, content substance cooled to a temperature below the congealing point of said capsule forming liquid, means for heating said capsule forming liquid before introduction into said vessel, and separate heating means in said vessel for counteracting the excess cooling effect of the content material.

12. In capsule forming apparatus, the combination comprising a vessel containing congealable capsule forming material in liquid condition, a plurality of orifices adjacent the bottom of said vessel for introducing globules of content substance into said capsule forming liquid, separate lines connected directly to each of said orifices and having refrigerating jacket means around the same for cooling the content substance to temperatures below the congealing point of the coating liquid, said jacket means extending into close proximity to said orifices.

13. In capsule forming apparatus, the combination comprising a vessel containing congealable capsule forming material in liquid condition, a plurality of orifices adjacent the bottom of said vessel for introducing globules of content substance into said capsule forming liquid, separate lines connected directly with each of said orifices for conveying content substance thereto, refrigerating means for said lines extending into close proximity to said orifices, and regulating means for the discharge of globules of content substance

75 from said orifices.

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14. Apparatus for fabricating capsules, comprising in combination a chamber having therein two superposed layers of liquids of different gravities, one of which liquids is a coating forming liquid, and a layer of liquid between the two aforesaid liquids and immiscible with both and of a gravity intermediate thereof to maintain said liquids separated.

15. In an apparatus for fabricating capsules, a vessel, means for introducing a capsule forming liquid into said vessel, heater means for maintaining the capsule forming liquid in heated condition in said vessel, a plurality of orifices in said vessel through which content substance may be introduced into the capsule forming liquid, a supply line for content substance individual to each orifice and extending directly thereto, means extending into proximity of said orifices for refrigerating said supply lines, a column of cooling liquid superposed in and being of lower gravity than the capsule forming liquid and through which capsules rise and float on the surface thereof, and means for removing capsules from the surface of the cooling liquid.

16. In an apparatus for fabricating capsules, a chamber having therein superimposed layers of

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capsule forming and cooling liquids of different specific gravity and immiscible with each other, a plurality of nozzles in said chamber for injecting content material into the capsule forming liquid, tube means for directing content material to said nozzles, and refrigerating means common to said tube means for cooling the content material passing therethrough, said refrigerating means extending into close proximity to said nozzles.

17. In an apparatus for fabricating capsules, a chamber having therein superposed layers of capsule forming and cooling liquids of different specific gravity and immiscible with each other, means for heating the capsule forming liquid within said chamber, a nozzle having its discharge end projecting into the layer of capsule forming liquid at a point above the bottom of such layer, tube means connected to the inlet end of said nozzle for directing content material thereto, and means for refrigerating the tube means for causing cooled content material to be discharged from said nozzle, said refrigerating means extending into close proximity to said nozzle.

ROBERT W. GUNNELL.