Jan. 6, 1953 F. E. STIRN 2,624,163

METHOD OF MAKING COLORED GELATIN CAPSULES

Filed April 22, 1948

Fig. 1.

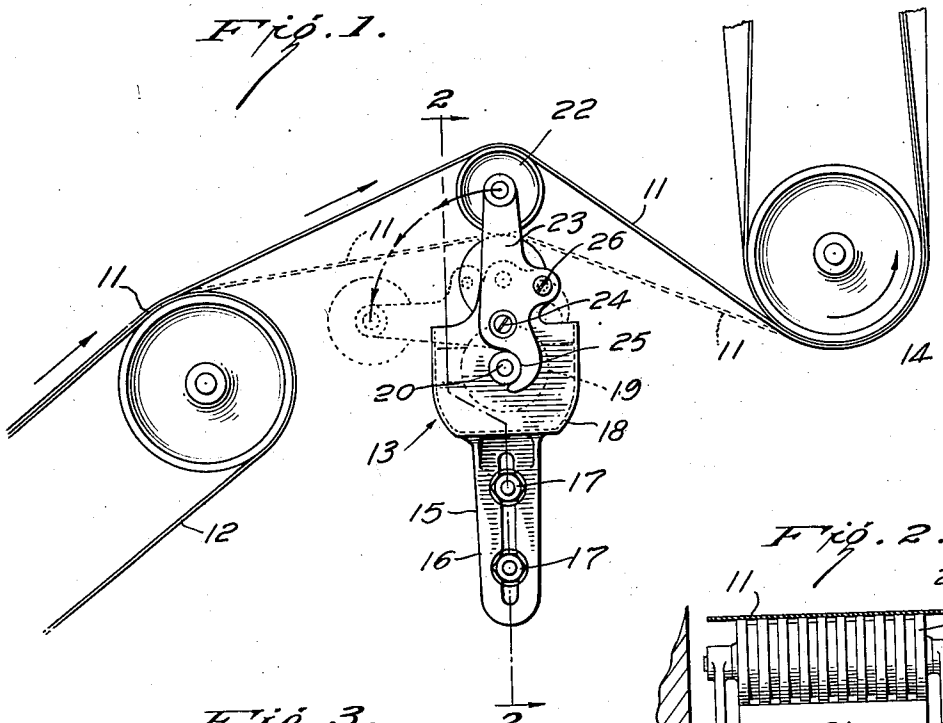

Fig. 2.

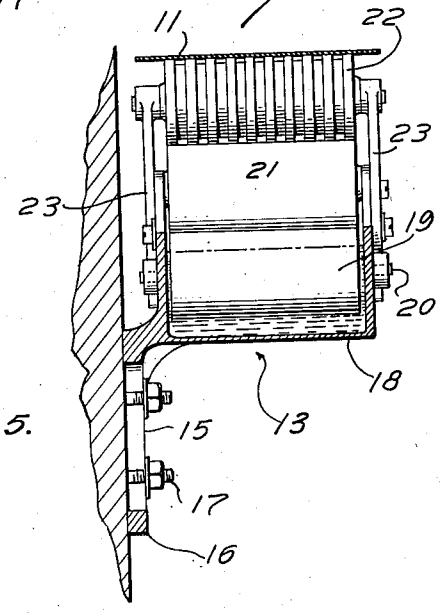

Fig. 3.

Fig. 4.

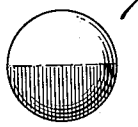

Fig. 5.

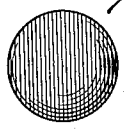

Fig. 6.

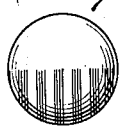

Fig. 7.

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METHOD OF MAKING COLORED GELATIN CAPSULES

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Application April 22, 1948, Serial No. 22,598

1 Claim. (Cl. 53—5)

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My invention relates to improvements in gelatin capsules, particularly those which are formed from continuous strips of gelatin film; pertaining more particularly to methods of coloring of at least part of the surface of such capsules in such areas as may be considered desirable.

In the past soft gelatin capsules have been produced from gelatin film by stretching the film to a desired form and encapsulating therein the desired contents. It has been customary to form such gelatin capsules in some instances, in clear, colorless gelatin and in others from colored or pigmented gelatin, as is desired for purposes of making the gelatin capsule more attractive. Such methods of coloration are described for example in the Patent No. 730,643 to Anthony M. Hance. Additional methods are disclosed in the Patent No. 1,134,156 to H. R. Planten, which again describes soft gelatin capsules being made from colored film, in this instance disclosing two different colors of film for different portions of the surface by making the capsule from two sheets of two different colors.

My invention relates to a very simple and more convenient method of forming capsules which comprises casting the gelatin in any convenient manner, of any convenient color, as may be desired, and treating the film after having been cast, but preferably before the capsule formation, with a soluble dye in a suitable solvent whereby the surface only of desired portions of the gelatin film is colored in accordance with the operator's choice to form capsules in which various portions may be colored. It is convenient by my method to form capsules in which the two different portions of gelatin strip from which the capsule is formed are differently colored, but additionally it is possible by my method to form capsules in which trade-names or identifying marks may be placed on the surface of the capsules for purposes of identifications of origin, giving information to the physician, or patient, or such other purposes as may be deemed desirable or necessary.

The methods of forming the gelatin film and the methods of encapsulating material in the formed films are not a part of the present invention and may be in accordance with conventional practice or in accordance with such novel procedures as are described in the patent application of Frank E. Stirn and Arthur S. Taylor entitled "Method and Machine for Making Capsules," Serial No. 16,554, filed March 23, 1948.

As described in that patent application gelatin films may be cast in which titanium dioxide pigment or other pigment is present to render the film opaque and in which a suitable dye may be dissolved so as to color the film throughout to a uniform density as may be desired. Additionally this base film may have a desired flavor or odor imparted thereto by the addition to the gelatin mixture of suitable essential oils or other aromatic materials.

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The method of treating such a film and further objects of my invention will become obvious from the following description, accompanying drawings and appended claims.

Figure 1 is a side elevation of a coloring roll stack which may be used in accordance with this invention to color a gelatin film.

Figure 2 is a front view of the roll stack, in section, showing the gelatin film passing thereover, taken along line 2—2 of Figure 1.

Figure 3 is a view of a contacting roll certain portions of the surface of which are raised to give selective coloring to the gelatin film.

Figure 4 is a view of a typical capsule showing one-half of the capsule being colored differently than the remainder.

Figure 5 shows a capsule, the entire surface of which is colored.

Figure 6 is a figure of a capsule showing a portion of the gelatin capsule which does not have a surface dye thereon, and the remainder dyed with a striped pattern.

Figure 7 is a gelatin capsule a portion of the surface of which is so colored as to show a desired inscription, as for example that produced by the roller of Figure 3.

Different methods of forming the gelatin film can be used in connection with my method of coloring. There is shown, accordingly, only a portion of the gelatin film 11, as it is fed from a conveyor feed belt 12 over a dyeing assembly 13 to a take-up roller 14. The conveyor feed belt and take-up roller are not part of this invention and are shown diagrammatically only as they may be any of those previously known.

For my purpose of dyeing the film it is necessary that a dye roll mechanism be used such that a sufficient and not excess portion of dye is continuously transmitted to the gelatin film continuously and uniformly.

In such traditional printing procedures as are used in printing paper webs, as for example a newspaper, it is customary to transfer ink from an inking roll to a type surface and from there to the paper, with the paper at the time of printing being pressed firmly against the surface of the type by some form of backing roll. Such a form of backing roll would not be satisfactory

with a gelatin film because the film is fragile and extremely sensitive to pressure.

Whereas a very light pressured guide roll or guide belt could be used to assist in positioning the gelatin film against the film contacting dye roll it has been found that by suitable choice of dye roll material, dye, and solvent therefore, it is possible merely to use the pressure of the gelatin film caused by its weight or the slight tension which is necessarily present in the film as it is handled to cause the gelatin film to rest against the surface of the roll sufficient for transfer of the dye. This is shown clearly in Figure 1 in which the dyeing assembly **13** includes a support frame **15** adapted to be suitably supported in spaced relationship to the film, preferably by adjustable means, as for example the support slot **16** and clamping bolts **17**. Supported thereby is the dye trough **18** adapted to contain the desired dye in a suitable solvent as later described. If desired it is within the scope of this invention to supply the dye trough **18** with a float system for a continuous level maintenance by means of an automatic system or with a supply tube and feed glass whereby the dye level may be maintained by the operator. Such dye level maintenance is not particularly critical and may be most simply controlled by the operator adding additional dye solutions as is required to the trough from such supply container as is convenient. Suitably journaled in the dye trough **18** is a partially submerged roller **19**, so called because a portion of the surface of this roller is submerged beneath the liquid level of the dye solution during a portion of its rotation. This roller may be journaled either on pinions, or a shaft, so as to be freely rotatable and so as not to bind from the action of the dye and its solvents.

For purposes of dyeing gelatin film it has been found that a Micarta roll journaled on a brass shaft is particularly suitable. The brass does not readily corrode. The Micarta roller slides freely upon the brass shaft **20** with a minimum of friction, is easily wet by the dye solutions, and transfers an adequate and effective but not excessive quantity of the dye as it is so rotated. The Micarta used may be prepared by molding a phenolformaldehyde type resin (Bakelite) with a linen or cotton fabric, in accordance with conventional practice. It is essential that the smooth gloss surface obtained from a well polished mold be broken; a convenient method of doing this is to grind the surface of the mold with an abrasive grinding wheel or to turn the surface of the roll as for example on a standard metal working lathe, whereby the surface of the roll is smooth and uniform but yet not glazed. Journaled above, parallel to, and in contact with the submerged roll is a dye transfer roll **21** which is suitably journaled on a shaft above and parallel to the submerged roll **19**. While not necessary, it may be desirable that the center to center distances of these rolls be adjustable so that the rolls may be turned down rather than replaced when worn.

As shown in Figure 1, there may be journaled above the dye transfer roll an embossed roll **22**. This embossed roll is supported by an embossed roll bracket **23** on each side, which in turn is supported by a bracket screw **24** about which it may rotate and which positioned by collar **25** may be clamped by a clamp screw **26**. As shown, the embossed roll may be clamped in a vertical position as shown in solid lines in Figure 1, or dropped to a non-functioning position as shown in dotted lines in Figure 1.

Both the dye transfer roll **21** and the embossed roll **22** may well be made from Micarta, although other suitable materials may be used. As shown in Figure 2, the embossed roll consists of a series of raised peripheral areas so that a series of parallel lines are dyed upon the surface of the film. A suitable inscription or legend may be dyed upon the surface of the film by the introduction of a suitably embossed rule as shown in Figure 3. Either the major or minor portion of the inscription may be the raised portion which transfers the dye to the surface of the gelatin film.

For best results it has been found most desirable that a dye rather than a pigment be used for the coloring of the surface and it has been found desirable that the dye used possess certain characteristics. For the purposes of human consumption it is essential that the dyes used be non-toxic. Dyes approved for food, drug and cosmetic use are particularly suitable because those approved by the Federal Drug Administration or other government agencies for use in food and drugs have been already checked by toxicity studies and additional studies are not necessary to permit their use for human consumption. Other dyes may be chosen for other purposes. If the capsules are used for other than human consumption a very broad choice of dyes may be made.

It is desirable that the dye solvent used be reasonably volatile, so as to insure easy removability; be water miscible, so that it will penetrate into the surface of the gelatin film and will not form a repellent layer thereon, be cheap, easily obtainable; and it must dissolve the specific dyes being used. For standard gelatin films it has been found that methyl alcohol and ethyl alcohol are particularly useful. Higher alcohols such as isopropyl, propyl, the butyls, benzyl, etc., may be used but because they are less volatile it is more difficult to remove them and because the higher alcohols are less water soluble there is more of a tendency for the dye not to transfer smoothly into the gelatin film and instead to remain encrusted on the surface. Polyhydric alcohols are in general not sufficiently volatile for commercial utilization. Acetone may be used but it has a tendency to dry too quickly and not give a sufficiently deep or clear print and seems to leave the dye mainly on the surface. Water, itself, does not dry sufficiently quickly and tends to run on the surface of the film giving feathering rather than clear lines. The lower alcohols, particularly methyl and ethyl, are the most satisfactory as they are good solvents for many of the drug and cosmetic dyes, they transfer reasonably clear to the surface of the gelatin film, do not encrust on the surface, and under normal conditions give a smooth, rapid and effective transfer which may in part be due to the rapid diffusion of the alcohol into the gelatin film.

Petroleum ether was used as a solvent but dried too quickly and did not appear to be sufficiently water miscible to transfer the dye into the gelatin film properly. It is operable, but not a preferred embodiment.

Example 1

Micarta rolls were used, the embossed roll was dropped to the inactive position. A solution was prepared containing 5% of the dye known as "D and C Violet No. 1" in methyl alcohol. The

dye trough was filled so that the submerged roll was nearly covered and the capsule machine operated with the gelatin film contacting the dye transfer roll 21. A smooth, uniform, even violet coloration was given to the surface only of the gelatin strip. The alcohol evaporated sufficiently rapidly so that no complications were encountered in the operation of the machine and capsules were produced with the entire surface dyed violet.

Example 2

The dye known commercially as "F, D and C Red No. 3" was prepared as a 5% solution in ethyl alcohol. The embossed roll 22 was raised to operating position, an embossed roll consisting of raised adjacent strips, as shown in Figure 2, being used. The capsule machine was operated, giving smooth, uniform, alternately colored strips on the surface of the gelatin film, and was so operated that only the gelatin film forming one-half of the capsule was colored by running only one-half the forming film over the dye roll. Smooth, evenly dyed bars were formed on the surface of the film with bright, sharp, clear edges. The dye was readily and evenly transferred to the surface of the film. Capsules of this type are shown in Figure 6.

Example 3

The dye known as "D and C Violet No. 1" was dissolved to the extent of 5% in water. The embossed roll was dropped and the device operated with the dye transfer roll in contact with the surface of the film. An even transfer of color took place but the additional water introduced on to the film caused the film to dry so slowly as to make it necessary to operate the capsule forming machine at less than the otherwise most desirable speed.

Example 4

A 1% solution in acetone was prepared of "D and C Violet No. 2," and the dye mechanism operated with the dye transfer roll contacting the surface of a single film. The acetone dried too quickly so the imprint was not as deep or as clear or as uniform as with alcohol as a solvent but a capsule which was commercially satisfactory was obtainable as illustrated in Figure 4.

Example 5

A 5% solution of "F, D and C Red No. 3" was dissolved in ethyl alcohol. An embossed roll with an inscription thereon as shown in Figure 3, was placed in the embossed roll bracket and raised to contact the surface of the film. A smooth, uniform dyed print was obtained on the surface whereby an inscription such as a name and trademark were smoothly embossed on the surface of the gelatin film forming capsules with the name of the contents dyed in the surface thereof.

Example 6

The machine was rearranged so that two independent coloring mechanisms were present, in the first of which was placed a 5% solution of "F, D and C Green No. 1" in ethyl alcohol, with the dye transfer roll in contact with the surface of the film. A short distance therefrom was placed the second dyeing assembly with an embossed roll containing a trade inscription raised on the surface thereof. This trough was filled with a 5% methyl alcohol solution of "D and C Red No. 28." A timing chain was connected to the embossed roll 22 so that it was driven in timed relationship to the capsule forming dies whereby capsules were formed with the trade insignia uniformly printed in red on a green background and accurately registered on the surface of the filled gelatin capsules.

Example 7

A 1% solution of "F, D and C Red No. 3" in ethyl alcohol was substituted but the apparatus otherwise operated the same as in Example 1. A rather light color coat was formed, but entirely uniform and satisfactory for purposes where pale colors are desired.

Example 8

A 1% solution of "F, D and C Green No. 1" was prepared in isopropyl alcohol and placed in the dye trough with the gelatin film contacting the transfer roll. The gelatin film was inverted by running over a pair of rolls having a half twist and an additional coloring mechanism was placed in contact with what was to form the internal surface of the capsule. In this trough was placed a solution of 5% "F, D and C Red No. 3" in tertiary butyl alcohol, with the strip then fed to the capsule forming machine. An embossed roll was used in this second machine with the name of the maker thereon. Capsules were formed with a comparatively smooth, uniform dye coat on the outside which effectively concealed the dye insignia on the inside of the capsules. The capsules could be identified as to origin by slicing them in two and reading the name of the maker on the internal surface.

In general, it will be found that the dyes will print evenly and uniformly. However, with certain gelatin formulations particularly those in which an unusually high percentage of a plasticizer such as glycerine is used, the dyes will exhibit a tendency to bleed after the printing, perhaps because such plasticizer acts as a solvent for the dye and permits it to run. Under such circumstances, a dye must be selected such that due to its lower solubility in glycerine it will not bleed to an undesirable extent. With the harder formulations, such as are preferred for the powder filled capsules, such troubles with bleeding are not normally encountered, and clear, sharp lines are the rule. If a high oil content is present in the capsule, with a high glycerine content, it will be found that the oil remains in place, but if a low glycerine mix is used, some of the oil may tend to penetrate the gelatin, and in such cases, it may be desirable to coat the inside of the capsule with an impervious coating such as a resin to prevent the oil from blurring the color lines.

Other dyes may be used. The publication of the Federal Security Agency, Food and Drug Administration, Service and Regulatory Announcements, Coal Tar Color Regulations list such acceptable colors. For those skilled in the art the dye index lists the colors of the dye, the solvents in which they dissolve, whether or not they are water compatible and their toxicity. Any dye which is acceptable from the standpoint of toxicity and which meets solubility characteristics is acceptable and may normally be readily selected from the dye indices without the necessity of experimentation. It is desirable that the dyes be at least somewhat water soluble so that they will go into the gelatin film more readily and will not tend to cake on the surface thereof. The number, the depth, and configuration of dye patterns may be varied in accordance with the choice of

the operator. The dyes on the capsules form particularly convenient methods of identifying the contents and the manufacturer according to predetermined codes and standards.

It is the intention of the inventor to reserve for himself and those in privity with him the distinctive colors of blue and yellow. Others may of course desire to use other color combinations.

It is convenient to use a gelatin film containing a pigment to prevent light from passing entirely through the capsule and to conceal the contents with a yellow dye in the base film and on the surface of which the desired patterns or insignia may be readily printed.

Whereas there has been described particularly the use of two and three roll mechanisms, to those in the art it will be obvious that four, five and six rolls could be used with the dye being transferred for the purposes of getting thinner coats. Additionally the roll stack could be arranged so that the final film contacting roll is above, below or at either side of the trough. Where several rolls are used or where the bearings are not substantially frictionless it may be desirable to have the film contacting roll power driven to prevent any pulling or slipping on the surface of the film. Whereas rolls of Micarta have been particularly described, rolls of soft wood, sintered glass, sintered metals, unglazed porcelain, well compacted felt, cloth, paper pulp, brass, bronze, stainless steel, etc., may all be used within the scope of my invention. Such rolls as type metal, steels, etc., must be clean so the ink will wet the surface to work properly. An off-set transfer roll may be used, as may lithographic rolls, if roll surfaces are selected that possess the well known requirements for these specific purposes.

Whereas in the past it has been necessary to use different gelatin supplies to get different colored films in the capsule, my method of coloring the film as cast insures that both gelatin films used for multiple colored capsules will possess essentially the same characteristics, as they are cast at the same time. The films will be homogeneous. It will be unnecessary to worry about having residue of one color left over and in general operating conditions will show the marked improvement that comes from simplified operations.

Having therefore set forth certain preferred embodiments, illustrating certain aspects of my improvements, as my invention I claim:

The method of forming an edible soft gelatin capsule with visible insignia which includes the steps of dissolving a non-toxic alcohol-soluble dye in a lower alcohol, transferring the thus formed solution from a bath to a pattern roll containing upstanding portions and marking a wet, freshly cast self-supporting soft edible gelatin film by contacting said film with upstanding portions of said pattern roll, thereby transferring portions of the solution to the desired portions of the wet, freshly cast, edible soft gelatin film, causing the alcohol to evaporate therefrom, and promptly forming edible soft gelatin capsules from portions of the thus marked strip.

FRANK E. STIRN.

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