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2,688,568

PROCESS OF PRODUCING DRAIN-CLEAR
CONTAINERS

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The present invention relates to drain-clear containers for liquids, particularly such that are provided on the inside surfaces with a silicone film. It concerns an improvement on such containers, whereby the silicone film is prevented from extending to the external surfaces of the containers where it manifests certain undesirable effects. The invention embraces a process for producing the improved, drain-free containers, preferably in sterile form.

Drain-clear containers provided with an internal silicone film are disclosed in the U. S. Patent No. 2,504,482 to Goldman. Owing to the water-repellent (hydrophobic) properties of the silicone film, such containers will promptly drain clear of any aqueous liquid contents, thereby reducing wastage, particularly in the case of aqueous-vehicle, liquid, pharmaceutical preparations.

A serious drawback of the known silicone coated containers is that the internal silicone film almost invariably extends to the outer surface where it interferes with the marking and labeling of such containers. The silicone film on the external surface is either formed unintentionally, when applying the internal coating, or else it is caused by "creeping" of the internal film to the exterior. It requires only traces of the silicone material to produce a highly water-repellent film of substantial area, so that it is well nigh impossible to avoid such external films, particularly when the desired internal films are applied by means of automatic machinery.

The marking or labeling of containers for liquid pharmaceuticals and chemicals is obviously of great importance. Hence, the drawback discussed above is indeed serious. It is referred to in the above mentioned U. S. Patent No. 2,504,482 where it is proposed to be overcome by prior application to the external surface of a protective layer of a substance which may subsequently be stripped off. This preventive measure is both awkward and expensive and hence impractical in most cases.

The primary object of this invention is to provide a simple and effective means whereby inadvertent application of a silicone film or the creeping thereof to the external surfaces of a container may be prevented. A further object is to produce sterile drain-clear containers having internal silicone films, which can readily be marked and labeled with the use of ordinary adhesives. Further objects and advantages will become apparent from the following detailed description.

According to the present invention the external

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surface of the container is provided with an exceedingly thin film of a water-soluble or dispersible, high molecular weight, organic, surface-active agent. Such film need only be monomolecular in thickness, so that a very small amount of the surface-active agent goes a long way. It has been found that such film will effectively prevent creeping of the internal silicone film and neutralize the water-repellent properties of any silicone film that might inadvertently have been formed thereunder. The exact mechanism whereby the undesired effects of the external silicone film are overcome is not known, but it is a fact that containers having such an external surface active film can readily be marked and labeled with the use of ordinary materials.

The internal silicone film may be applied in a variety of ways. Some of these are disclosed in the above mentioned Goldman patent. Other methods for applying such water-repellent films are shown in the U. S. Patents 2,306,222 to Patnode, 2,386,259 to Norton and 2,439,689 to Hyde. The preferred method is to apply a dilute aqueous emulsion of the silicone to the interior of the container, while it is inverted, and then to dry the residual coating.

The term "silicone" as used herein refers to the monomeric and polymeric organo-siloxanes in which the organo radicals are connected by carbon-to-silicon bonds, while the silicon atoms are connected to oxygen atoms. Such silicones are disclosed, for example, in the U. S. Patents Nos. 2,258,218-2,258,222 to Rochow, and the polymeric forms are described in the Goldman patent. Generally, they represent oily to resinous substances. Various types are now available on the market in water-emulsifiable form.

The surface-active external film is generally applied by treatment with a dilute, aqueous solution or dispersion of the surface active agent, followed by drying. Inasmuch as said agent is water-soluble or dispersible, this film can be applied prior to the internal silicone film only when the latter is applied in such a manner that no aqueous solvent comes into contact with the external film. Generally, it is preferable to apply the external film after applying the internal silicone film. This external film will effectively prevent creeping of the internal silicone film. Furthermore, it appears that the water-repellent properties of such traces of silicone as may inadvertently have been applied externally, are neutralized by the subsequently applied external surface active film.

The water-soluble or dispersible, high molecular weight, organic, surface active agents suitable for forming the external film are available in a great variety of types. Any of the well-known detergents, and dispersing or emulsifying agents may be employed. There may be mentioned for example the usual soaps consisting of alkali metal salts of higher fatty acids, as well as of other high molecular, organic acids, such as abietic acid; furthermore, sulfated long chain alcohols, sulfonated oils, aromatic sulfonates, quaternary ammonium salts, fatty acid esters of polyvalent alcohols, their ethylene oxide derivatives, etc. As pointed out previously very small amounts of these surface active agents are sufficient for the purposes of the invention, since the external film formed thereby need be only monomolecular in thickness. A high concentration of the surface active agent in the treating liquid does not materially improve the results.

In its preferred embodiment the invention is applied to glass containers for aqueous-vehicle, liquid pharmaceutical preparations of the type shown in the Goldman patent. However, it is obvious that many other applications of the invention are possible. The container may be composed of metal or of a glazed ceramic material. Broadly speaking, the invention is applicable to all containers for aqueous liquids, to which it is desired subsequently to apply labels or other external markings. In the preferred embodiment of the invention the preparation of the pharmaceutical container is combined with the usual dry heat sterilization. The process of the invention may be carried out in the apparatus available for washing and sterilizing glass containers, such as pharmaceutical containers and milk bottles, with a minimum of adjustment. The preferred sequence of steps is to rinse the container internally with a dilute solution or dispersion of the silicone, then to rinse the container externally with a dilute aqueous solution or dispersion of the surface active agent, these steps being carried out while the container is inverted, so that excess liquid will drain off, and finally to dry the thus treated container, preferably at temperatures high enough and for a sufficient period to effect sterilization. It is, of course, possible to carry out the internal and external rinsing operations simultaneously, but this will interfere with the recovery of the excess treating liquids.

After the containers have been treated as outlined above, they may be stored under sterile conditions, until they are filled. After filling and closing it will be found that the usual labels or other marking materials can be applied to the external surface without any difficulty arising out of the creeping or inadvertent application of a silicone film. This constitutes a great advantage over the containers that lack the external surface active film of the invention.

The following detailed examples of how the invention may be practiced are given merely for purposes of illustration. Obviously, the procedure, apparatus and materials used in these examples can be varied widely.

Example I

Clean 10 cc. glass bottles of the type used for packaging solutions or dispersions of penicillin salts are placed over a series of spikes on a traveling belt of a bottle washing machine. The individual bottles are inverted with the spikes entering through their mouths. The spikes are provided with openings through which a rinsing

liquid can be forced into the interior of the bottles.

In the first stage of the treatment a rinsing liquid consisting of a 1% aqueous emulsion of a silicone oil available on the market (product of the Dow-Corning Corporation) is injected through the spikes to thoroughly coat the interior surface of the bottles. Excess rinsing fluid drains into a tank below the belt and is reused. The belt then proceeds to the second stage of the treatment where the bottles are thoroughly washed from above by streams of a dilute solution of a soap (product of Lightfoot-Schultz Company). Again the excess treating liquid drains into a tank below the belt and may be reused.

In a third stage they are removed from the spikes and placed in suitable containers or racks. Such containers or racks are placed in drying and sterilizing ovens where they are subjected to temperatures of from 170° C. to 250° C. for periods of from 2 to 4 hours. This treatment thoroughly dries and fixes the films, destroys pyrogenic materials and effects a complete sterilization. The sterile bottles are then stored under sterile conditions until they are filled with the penicillin solutions or dispersions. After stoppering the bottles they can be labeled or marked externally without any difficulties arising out of inadvertent presence on the external surfaces of traces of silicone.

Example II

The process described in Example I was repeated, but in place of the dilute soap solution, a solution of 0.2% concentration of keryl benzene sodium sulfonate was used. The resulting bottles were much the same in quality. They could be labeled readily, and there appeared to be no silicone on the outer surface.

Example III

The process described in Example I was repeated using a 1.0% solution of a polyoxyethylene derivative of sorbitol mono-oleate in place of the dilute soap solution. The bottles obtained in this manner were shown to be particularly useful. When they were filled with an aqueous suspension of a pharmaceutical such as procaine penicillin the suspension readily drained from the walls of the container. Labels were applied to the outside of some of these bottles with no difficulty.

Example IV

The process described in Example I was repeated with the use of a 0.2% solution of stearyl dimethyl benzyl ammonium chloride in place of the dilute soap solution. Here again the bottles prepared in this manner were shown to be excellent drain-clear containers and their outer surfaces appeared to be free of silicone so that labels of various types could be readily applied thereto.

The silicone rinsing emulsion may be stabilized by the addition of small amounts of a dispersing agent, such as morpholine oleate or sodium lauryl sulfate. There is little danger of separation due to the fact that this emulsion is constantly being agitated in use and recirculation. Aqueous emulsions are preferred over solutions in organic solvents, as described in the Goldman patent, because such organic solvents are more expensive than water and subject to a higher rate of evaporation with the production of undesirable fumes.

As many apparently widely different embodiments of this invention may be made without departing from the spirit and scope hereof, it is to

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be understood that this invention is not limited to the specific embodiments hereof except as defined in the appended claims.

What is claimed is:

1. A method for preparing a liquids dispensing container which is clear-draining interiorly and to which external markings and labels may readily be applied, which comprises wetting the internal surfaces of the container with an aqueous emulsion of a silicone oil, washing the external surfaces, prior to drying of the silicone, with a dilute aqueous solution or dispersion of a high molecular weight, organic detergent, whereby creeping of the silicone to said external surfaces is effectively prevented, and then drying the container.

2. A method as claimed in claim 1, wherein the

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container is maintained in an inverted position during the liquid treating steps, while the silicone emulsion is injected interiorly from below and the detergent solution applied exteriorly from above, and the drying step is carried out at temperatures sufficiently elevated to effect sterilization.

References Cited in the file of this patent

UNITED STATES PATENTS

Number	Name	Date
2,306,222	Patnode	Dec. 22, 1942
2,395,654	Brannigan	Feb. 26, 1946
2,504,482	Goldman	Apr. 18, 1950
2,557,983	Linder	June 26, 1951