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2,880,901

CONTROLLED TORQUE GASKET COMPOSITIONS

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The present invention relates generally to improved gasket compositions in the form of permanent rubbery gels of the type normally referred to as "plastisols," the improved compositions being adapted for use with rotatable caps to provide the same with controlled opening torques following application to glass containers in sealed relation therewith. More specifically, the invention is directed to the provision of a viscous coating on the sealing surface of the "flowed-in" plastisol gasket material carried by the cap, which coating is capable of internal shear upon the application of a cap-removing torque of predetermined magnitude to prevent seizure between the plastisol gasket material and the glass finish of the jar following application of the cap to the jar in accordance with known automatic capping procedures.

The rotatable closure cap with which the improved plastisol gasket material of the present invention is particularly adapted for use is of the type disclosed in a copending application, Serial No. 520,881, filed July 8, 1955, now Patent No. 2,841,304, granted July 1, 1958. This type of cap is formed from a metal shell having a top panel portion and a depending skirt portion, the outer margin of the inner surface of the top panel portion being provided with a "flowed-in" plastisol gasket material into which is pressed the top rim or edge of a glass container when the cap is forced down into sealed relation therewith. The depending skirt portion of the cap is provided with inwardly directed lugs which are slidingly moved along glass threads or lugs formed in the outer surface of the container inwardly of the top edge thereof. The cap is rotatably advanced onto a container in a known manner and the advancing of the top panel of the cap toward the top edge of the container brings the gasket in to intimate sealing relation with the top edge of the container to seal the same. In sealing glass containers the contents placed in the containers are frequently at elevated temperatures and the cap including its plastisol gasket is also frequently heated when applied to the container.

The lastisol gasket material in use at the present time is made from a paste-forming resin-plasticizer mixture which may contain a filler. The mixture, upon fluxing at elevated temperatures after being applied to the inner surface of the top panel of the cap, forms a permanent gel which becomes elastic and rubbery upon subsequent cooling. Upon the reheating of the plastisol gasket material, during sealing operation requiring heat, when the cap carrying the same is brought into contact with a filled heated jar, the plastisol loses its elasticity until subsequently cooled at which time the original elasticity is quickly regained. Still further, a plastisol of this type is known to relax under pressure during cooling. These two inherent physical phenomena of plastisol gasket material combine to make it commercially difficult if not impractical to apply caps to jars without either applying the caps too loosely so that they become "leakers" or too tightly so that they "freeze" on and require excessive opening torque to remove.

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In order to maintain the seal, hermetic or otherwise, there must be sufficient friction to prevent a rotatable cap from backing off. Since there is very little friction between the metal lugs or threads and the glass lugs or threads, the ability of a cap to stay on is due primarily to the friction between the gasket compound and the glass. Therefore, while there must be sufficient initial friction to prevent a cap from backing off, the friction must not increase with time by seizing so that the cap is difficult to remove.

It was found that the friction caused by seizing developed between known plastisol gasket material and the glass finish increases substantially upon standing. Consequently, the torque necessary to initially break the seal between the cap and the container may increase several times. Opening torques ranging from 3 to 28 inch-pounds have been measured within 5 to 10 seconds following the capping operation with certain caps. After a little time as 1 hour, the opening torques were sometimes as high as 80 inch-pounds. Longer times have been known to require as high as 100 inch-pounds. As such tests revealed, the values of the opening torque subsequent to the capping operation were found to be extremely variable and difficult, if not impossible, to control. Ideal opening torque for rotatable caps is generally considered to be within a range from about 20 to 35 inch-pounds regardless of the time elapsed following the capping operation. Using the principle of this invention a formulation, given later, has been made and lined into large caps (77 mm. dia.) which were sealed onto jars. Maximum opening torque requirements were 35 inch-pounds. This approaches the ideal range even though a large size cap was used. This range of opening torque is such that there is sufficient adhesion to prevent the cap from backing off out of sealing engagement with the container and from becoming accidentally displaced in handling, and yet it may be readily overcome without difficulty upon intended removal of the cap from the container.

Plastisol gasket material formed from resins such as polyvinyl chloride exhibit good initial elasticity when cold but quickly lose this elasticity under constant deformation while sharply losing the same when heated to around 190° F. Elasticity is sharply regained upon cooling and the original properties reassert themselves. While the compressive force exerted against the gasket material will cause the same to relax upon cooling, the degree of relaxation is not sufficient to counterbalance the adhesiveness caused by the elasticity being quickly regained. Consequently, the compressive force developed by the regaining of elasticity greatly exceeds the reduction in compressive force caused by relaxation during the cooling of the plastisol gasket material.

With increased compressive force between the plastisol gasket and the glass finish, seizure or friction increases correspondingly. Thus, upon standing, the torque necessary to rotatably remove the cap from the container increases proportionately with increased seizure or adhesiveness. This is true also when the cap is not heated, but the opening torques do not reach quite as high a value, and it takes more time for the highest value to be obtained. During the capping operation the plastisol, being in a heated condition, exhibits its minimum adhesiveness. Consequently, it is necessary to rely at this phase of the operation on the friction developed by the torque applied in rotating the cap on the container. This torque must be adequate in magnitude to establish sufficient friction between the cap and container to prevent backing off of the cap following the capping operation until sufficient adhesiveness is established between the plastisol and the container by the regaining of elasticity upon cooling. This is not a

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problem in cold sealing, but controlling the tightening torque is very important. As previously described, heretofore it has been found impractical, if not impossible, to control the friction developed between the gasket and the container due to the sharp regaining of elasticity of the plastisol upon cooling. Consequently, there is no known procedure which may be followed to produce capped containers, the caps of which may be removed from the containers by the application of opening torques falling within a specified, relatively limited range.

It is during the first minute or so following the heated capping operation that adhesion is begun. The surface of gasket material contacting the glass finish is somewhat wet due to the plasticizer migrating when the plastisol is heated. During the first minute of cooling the plasticizer is re-absorbed by the plastisol while the latter is returning to its completely solid state. The fast regaining of elasticity during this time materially increases the pressure being applied by the plastisol against the container. This adhesive force continues to build up even though there is a continual drop in force due to relaxation, the latter drop being incapable of balancing the former increase.

The seizing is considered to be due to the turning of the slightly liquid phase caused by the migrated plasticizer into a solid phase at a time when compressive force is increasing rapidly as a result of sudden regaining of elasticity. These conditions are ideal to promote gluing as it has been found that wax blooming to the surface and solidifying during the cooling of a sealed package results in intimate filling of the voids between the surfaces under seal. This solid interface must be broken by the use of physical force to open the package and the requisite force is substantial due to the necessity of breaking a solid. The same problem is believed to exist where the liquid phase returns to a solid phase on the surface of the gasket during cooling of the same. Thus, the simultaneous solidifying and regaining of elasticity, the latter resulting in an increase in compressive force, promotes seizing which may be of a magnitude requiring the application of a substantial opening torque to overcome.

It is an object of the present invention to provide improved plastisol gasket compositions which, when applied to a cap in accordance with known practices and the cap applied to a container by widely used forms of automatic capping apparatus, provide a controlled frictional seal between the gasket material and the container which may be overcome by the use of a torque falling within a predetermined limited range at any time following the capping operation regardless of the compressive forces developed by the gasket composition as a result of the regaining of elasticity upon standing.

Another object is to provide an improved sealing gasket composition of the plastisol type which exhibits controlled seizure upon use in a known manner in sealing a capped container, the frictional forces developed between the cap and the container being capable of predetermination to allow the cap to be removed at any time subsequent to the capping operation upon the application of an opening torque which falls within a relatively limited range, the controlled opening torque resulting from the presence of a viscous liquid interface between the gasket composition and the container, which interface is capable of internal shear upon application of the opening torque of specified magnitude.

A further object is to provide improved plastisol gasket compositions of the type formed from a paste-forming resin-plasticizer mixture containing discrete particles of plasticity-imparting filler material, the mixture being improved by the inclusion of liquid polybutene, the resulting mixture being transformed into a rubbery gel upon cooling following fluxing of the same, the polybutene migrating to the surface of the gel during fluxing

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and subsequent reheating of the same to provide a viscous liquid coating capable of internal shear at a predetermined torque following the application of a cap carrying the gasket composition in sealed relation with a glass container.

Other objects not specifically set forth will become apparent from the following detailed description of the present invention.

A plastisol of the type discussed above is in the form of a congealed paste-forming resin-plasticizer mixture containing in uniform distribution, finely divided discrete particles of a plasticity-imparting filler material and finely divided discrete particles of a paste-forming synthetic resin suspended in a non-volatile plasticizer for the resin, the resin being paste-forming with the plasticizer at a temperature below the fluxing temperature of the resin-plasticizer component and soluble in the plasticizer upon heating to the fluxing temperature. This type of plastisol forms at the resin-plasticizer fluxing temperature a permanent gel which becomes elastic and rubbery upon subsequent cooling.

To the original mixture of ingredients forming the plastisol, a quantity of viscous liquid polybutene is added which, upon the formation of the gel following fluxing of the mixture, being incompatible with the plastisol, provides an outer coating of viscous liquid which is capable of internal shear and, due to its viscosity, its further capable of supplying the necessary tack or adhesiveness to resist backing off of the cap following the capping operation. The heating of the mixture including the polybutene to fluxing temperature causes the polybutene to migrate to the surface of the plastisol and remain thereon to provide the same with a viscous liquid coating. Upon application of the cap to the container during the capping operation the polybutene coating provides a permanent interface between the plastisol gasket material and the glass finish and exhibits sufficient adhesiveness to prevent the cap from backing off while the plastisol is regaining its elasticity during cooling. Even subsequent to the full recovery of the elasticity and accompanying compressive force of the plastisol, the polybutene interface remains between the plastisol and container and upon removing the cap from the container it is necessary to merely apply a torque adequate to promote internal shearing in the polybutene interface and overcome the friction existing between the lugs of the cap and the threads of the container. Thus, while the friction existing between the lugs of the cap and the threads of the container is controlled to a predetermined extent by the capping operation, the frictional forces existing between the plastisol gasket material and the glass finish is controlled to a predetermined extent by the viscosity of the polybutene interface and the opening torque is the sum of these predetermined forces and consequently may be predicted and controlled to fall within an ideal opening-torque application range.

Polybutene not only exhibits the desirable property of controlling friction but still further meets many other requirements of a material used on food containers. Polybutene is non-thermosetting, non-toxic, permanently fluid, non-drying, has high electrical resistivity and is impermeable to gases. The polybutene is incompatible with the ingredients of the plastisol and migrates, bleeds or blooms outwardly of the same upon heating to cover the surface of the gasketing material. The coating formed by the polybutene prevents undue seizure due to its ability to internally shear when subjected to an opening torque. Migration to the surface is promoted during the fluxing of the plastisol composition to form the same into a permanent rubbery gel following the application of the mixture, using the "flowed-in" method, on the inner surface of the top panel of the cap. Subsequent reheating of the plastisol will cause additional polybutene migration to the surface. Due to continued tendency of migration of the polybutene, the cap may be stored indefinitely and still retain an adequate interface.

In forming the plastisol, a polyvinyl chloride resin is preferably used; however, this resin might be modified with a material such as polyvinylidene chloride or polyvinylacetate. The resin forms a paste with the plasticizer and is added thereto in the form of finely divided discrete particles and uniformly distributed in suspension in the plasticizer. Any suitable plasticizer may be utilized as long as it is non-volatile and a plasticizer such as Paraplex G-62 (a polyester epoxide made by Rohm and Haas, Philadelphia, Pennsylvania) has been found preferable. A filler material may be added in the form of finely divided discrete particles and is uniformly distributed throughout the mixture. A filler material such as barytes has been found to impart desirable flow properties to the mixture. The resin used should be substantially insoluble in the particular plasticizer at atmospheric temperatures while being completely soluble in the plasticizer at elevated fluxing temperatures.

On the basis of 100 parts by weight of resin, the plasticizer used will preferably fall within the range of 50 to 250 parts by weight. The filler material is preferably added in quantities within the range of 0 to 250 parts by weight. To the resin plasticizer mixture, which is formulated in any known manner, is added a quantity of liquid polybutene which will preferably fall within the range of 5 to 40 parts by weight. The molecular weight of a liquid polybutene is generally within the range of from about 400 to 1,500. The range of molecular weight preferred is from about 700 to 1,200. The particular molecular weight of polybutene utilized may vary considerably while attaining the desired results and with higher molecular weights the polybutene will exhibit greater resistance to internal shear to an extent that an opening torque of slightly greater magnitude is necessary to rotatably remove the cap from the jar.

Upon complete mixing of the resin-plasticizer mixture including the polybutene, the paste-like composition formed is readily adapted for application to the inner surface of the top panel of the cap to form an annular gasket of the type disclosed in the copending application identified above. The particular manner in which this paste-like mixture is applied to the cap is well known and is generally discussed in the copending application. The mixture exhibits adequate tackiness to adhere to the enameled surface of the cap and be retained thereby in its operative position during subsequent fluxing procedures at which time the mixture is transformed into a permanent rubbery gel. The fluxing of the resin-plasticizer mixture is completed when the plastisol reaches a temperature of approximately 350° F. The polybutene is not affected by the heat but, due to its incompatibility with the components of the resin-plasticizer mixture, will tend to migrate or bleed to the surface of the gasket ring carried by the cap and remain thereon even following subsequent cooling of the finished gasket and cap. The surface coating of polybutene being viscous and tacky cannot be easily removed from the surface of the gasket material and subsequent storage over protracted periods of time will not result in a reduced efficiency of the cap when the same is ultimately used in sealing a container.

While the above description deals with the use of liquid polybutene, it is believed obvious that any viscous liquid polymer may be used which is capable of internal shear and is incompatible with a plastisol composition of the type described so as to migrate to the surface of a plastisol composition during the fluxing thereof or during subsequent reheating. Still further, the polymer to be suitable for use in the manner described must be non-thermo-setting, non-toxic, permanently fluid, non-drying, and highly impermeable to gases.

The following examples illustrate typical plastisol compositions to which various polymers of polybutene have been added and further illustrate the use of the modified plastisol compositions in forming gasket material applied to the inner surface of a cap for use in

accordance with the principles of the present invention. The particular ingredients and amounts used as well as the conditions and procedures described below are merely illustrative and are not intended to limit the scope of the present invention.

	Parts by weight	Parts by weight
Polyvinyl chloride.....	100	100
Plasticizer (Paraplex G-62).....	50	80
Stabilizer.....	1	-----
Polybutene.....	20	35
Paraffin wax.....	3.4	-----
Filler (barytes).....	-----	200
	174.4	415

In the above formulations the stabilizer used is Ferro 707-X, a stabilizer soap made by Ferro Chemical Company. While this particular stabilizer is preferred, calcium ricinoleate may be used. While the use of Paraplex G-62 is preferred, Citroflex A-4, which is an acetyltributyl citrate, is an example of another plasticizer suitable for use in plastisol formulations of the present invention.

Obviously many modifications and variations of the invention as hereinbefore set forth may be made without departing from the spirit and scope thereof, and therefore only such limitations should be imposed as are indicated in the appended claims.

We claim:

1. A gasket composition adapted for use with a rotatable cap to provide the same with a controlled opening torque, said composition including a plastisol mixture having as its principal ingredients a finely divided polyvinyl chloride base resin and a resin plasticizer, to which mixture is added a liquid polybutene which is incompatible with the plastisol.

2. A gasket composition adapted for use with a rotatable cap to provide the same with a controlled opening torque, said composition including a plastisol mixture having as its principal ingredients a finely divided polyvinyl chloride base resin and a resin plasticizer, to which mixture is added a liquid polybutene which is incompatible with the plastisol during fluxing of the mixture to form a permanent rubbery gel and which upon cooling has a surface rich in polybutene.

3. A gasket composition adapted for use with a rotatable cap to provide the same with a controlled opening torque, said composition including a plastisol mixture having as its principal ingredients a finely divided polyvinyl chloride base resin and a resin plasticizer, to which mixture is added a liquid polybutene which is incompatible with the plastisol upon heating, the resin being paste-forming with the plasticizer at a temperature below the fluxing temperature of the resin-plasticizer component and soluble in the plasticizer upon heating, the molecular weight of the polybutene being within the range of from about 400 to 1,500.

4. A gasket composition adapted for use with a rotatable cap to provide the same with a controlled opening torque, said composition including a plastisol mixture having as its principal ingredients a finely divided polyvinyl chloride base resin and a resin plasticizer, to which mixture is added a liquid polybutene which is incompatible with the plastisol upon heating, the resin being substantially insoluble in the plasticizer at atmospheric temperatures but being completely soluble in the plasticizer at an elevated fluxing temperature at which temperature the plastisol is formed into a permanent gel which becomes elastic and rubbery upon subsequent cooling, the polybutene bleeding from the mixture during the fluxing thereof to permanently provide the composition with a surface coating of viscous liquid capable of internal shear.

5. Gasket material adapted for use with a rotatable cap to provide the same with a controlled opening torque

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when applied to a glass finish, said material being basically a fluxed plastisol in the form of a rubbery gel having as its principal ingredient a polyvinylchloride base resin-plasticizer mixture, the surface of said material adapted for sealing application to a glass finish being provided with a viscous liquid coating of polybutene capable of internal shear upon the application of a torque of predetermined magnitude.

6. Gasket material adapted for use with a rotatable cap to provide the same with a controlled opening torque when applied to a glass finish, said material being basically a fluxed plastisol in the form of a rubbery gel having as its principal ingredient a polyvinylchloride base resin-plasticizer mixture, the surface of said material adapted for sealing application to a glass finish being provided with a viscous liquid coating of polybutene capable of internal shear upon the application of a torque of predetermined magnitude, the polybutene being compounded in the resin-plasticizer mixture prior to the fluxing of the same into a permanent rubbery gel and being incompatible with the mixture and gel after the heating thereof.

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7. A rotatable cap adapted for application to a container in sealing relation therewith, said cap carrying a plastisol sealing gasket having as its principal ingredients a polyvinyl chloride base resin and resin plasticizer with the outer surface of said gasket being adapted for frictional sealing engagement with a surface of said container, said outer surface being provided with a coating of viscous liquid polybutene.

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