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54 Methods for forming sealing gaskets for containers.

57 A sealing gasket is formed on the inner surface of a screw closure for a container by application of a molten composition comprising thermoplastic polymeric material and slip aid and optionally also containing wax and/or plasticising resin. The method is of particular value in the formation of gaskets for plastic screw caps for pressurised containers.

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METHODS FOR FORMING SEALING GASKETS FOR CONTAINERS

It is standard practice to provide a sealing gasket on the inner surface of a closure for a container so as to improve the seal between the closure and the container. The gasket is usually formed by applying fluid composition to the closure and then solidifying the composition. Many of the compositions are applied as solutions or plastisols but these can incur the disadvantage that it is necessary to cure the material in the fluid and this in turn generally requires heating of the closure. This necessitates the provision of suitable ovens or other heating apparatus and if the closure is of a heat sensitive material, for example a thermoplastic, the heating may result in serious distortion of the cap.

It is known that, in theory, gaskets can be formed from molten thermoplastic composition merely by allowing the composition to cool and solidify against the inner surface of the closure. The thermoplastic composition can be introduced in solid form and then heated (with the disadvantages discussed above) or can be introduced as a melt and solidified onto the closure.

Although melt application is very desirable for some purposes, e.g. for lining plastic caps, the sealing properties of the resultant gaskets tend to be unsatisfactory, especially when the contents of the container are to be pressurised and the closure is a screw closure. For instance a pressure-tight seal may not be obtainable or may be obtainable only under such a high loading on the closure that it is impossible to remove the screw closure by hand,

In British Patent Specification No. 1092161 various compositions are proposed containing from 16 to 92 parts by weight of specified ethylene vinyl acetate copolymer

and 5 to 75 parts by weight of a specified compatible resin. The amount of resin is, in the examples, always between about 15 and about 50% by weight of the total composition. In example 6 it is stated that long skirt
5 tin plate crowns retain pressure in excess of 12 kg/cm^2 . However such crowns would not be removed by screwing and so the pressure retention can be achieved simply by increasing the loading on the closure during closing. In example 7 removal torques of 6 to 23 cm kg were
10 obtained for screw plastic and metal caps but in this example the container is not pressurised and when it contains soft drinks it is stated that the drinks are non-carbonated.

This is consistent with our experience, which is that
15 it is not possible, using existing thermoplastic melt compositions, to obtain good pressure tightness at an acceptably low removal torque.

In European Patent Publication 0031673 a hot melt adhesive is used for forming a sealing gasket and is
20 applied in foamed form. The foaming is said to result in the gasket having greater resiliency and requiring less material for its formation. However it incurs the disadvantages that it necessitates the provision of material to foam the melt and we find that foamed systems
25 only give adequate pressure-tight seals at unacceptably high removal torque values.

In the invention a sealing gasket is formed on the inner surface of a screw closure for a container by application to the inner surface of an unfoamed molten
30 composition that comprises 55 to 99% by weight thermoplastic polymeric material, 0 to 30% of a wax, 0 to 20% plasticising resin and 0.1 to 10% slip aid and has a viscosity at 172°C measured at a shear rate of 237 sec^{-1} of from 50 to 400 poise and that when applied as a melt
35 to a closure to form a standard gasket, as herein defined

provides a gasket that has a venting pressure, as herein defined, of at least 10.5 kg/cm^2 and that has a removal torque, as herein defined, of from 6.5 to 19.5 cm kg.

Preferably the gasket that is formed in the process of the invention does have the defined venting pressure and removal torque. Preferably the composition is applied, often at a temperature close to the quoted value (e.g. within 30% of it), onto a closure while it is spinning, e.g. at 300 to 8000 rpm.

10 The percentages quoted herein for the amounts of components of the composition are all, unless otherwise stated, expressed as percentages by weight based on the weight of fusible organic components in the composition.

The composition generally consists only of fusible organic components but if desired may include non-fusible organic or inorganic materials such as titanium dioxide, china clay or other inorganic pigments or fillers. If titanium dioxide is present its total amount is normally below 10% and most usually below 5%, for instance 0.5 to 20 3% by weight. Other materials may be used in place of some or all of the titanium dioxide in quantities such that the total amount of non-fusible organic and inorganic material is normally below 20%, usually below 10% and preferably below 5%

25 If slip aid is included in plastisol compositions the normal amount that is added is around 1% since it is accepted that very small amounts are sufficient to give optimum attainable properties and that there is no advantage in increasing the amount. In Specification 30 No. 1092161 the amounts of slip aid used in the examples range between about 8% and 18%. In the invention we never use more than 10% and generally the amount of slip aid is 1.5 to 5%.

We have surprisingly found that increasing the amount of slip aid by quite a small amount above the low amount

that would normally be regarded as adequate gives a dramatic improvement in venting pressure and removal torque properties. The precise amount will depend upon the particular type of slip aid and upon the other
5 components of the composition. It should therefore be determined by formulating a composition free of slip aid and then testing the effect of varying amounts of slip aid in that composition. This testing can be conducted over a narrow range of slip aid amounts, generally 0.2 to
10 3 or at the most 5%. The variations in properties within this range are far in excess of the routine variations that one would normally expect if the amount of slip aid is varied above, say 1%.

The reason for this great sensitivity, and the reason
15 for obtaining such good venting pressure and removal torque values upon optimising the amount of slip aid at a low level is not clear but may be associated with the fact that the composition does consist of a large amount of thermoplastic polymeric material (or thermoplastic
20 material plus wax) and is free of solvents, liquid plasticisers and other diluents and generally contains, at most, quite a low amount of plasticising resin.

The composition should have a viscosity within the defined range since outside these values it is difficult
25 to form, by melt application, a gasket having the desired properties. It is generally preferred that the viscosity, measured at 172°C at a shear rate of 237 sec^{-1} , should be below 300 and preferably below 200 poise. This facilitates the application of the melt
30 composition onto the screw closure. It is also generally preferred that the viscosity should be at least 90, and preferably at least 100 poise, since these higher viscosities seem to be associated with improved venting pressure and removal torque values. Preferably the
35 viscosity is between 110 and 180 poise, most preferably

120 to 170 poise with best results generally being obtained at values of 130 to 160 poise.

The venting pressure and removal torque values are determined on a standard gasket. This is a gasket
5 obtained by extruding the composition at 175°C through a nozzle onto the inner surface of a 28 mm diameter polypropylene screw cap. If an annular gasket is required, the nozzle directs the composition to one side of the cap while the cap is spinning at about 3,500 rpm.
10 The gasket weight is then 200-250 mg. If an overall gasket is required the nozzle directs the composition to the centre of the cap while the cap is rotating at about 5500 rpm. The gasket weight is then 350-400 mg.

Venting pressure is recorded using the Owens-Illinois
15 Secure Seal Tester. A polyethylene terephthalate bottle neck is fitted by a pressure-tight fixing to a bottle base in the Tester, so as to form a bottle. The lined cap is screwed onto the test bottle by a conventional capping machine set at a head load of approximately 40 kg
20 and a closing torque of 10-15 cm kg.

A compressed air line leads to a needle fitted in the bottle base so as to permit compressed air to be forced into the bottle. The bottle, including the screw cap, is immersed in water and the water is observed, as the
25 pressure in the bottle is increased, for the escape of air from the cap. Air pressure is gradually increased stepwise at a controlled rate. The pressure is released 10 seconds after 14 kg/cm² has been reached. If venting from the cap is observed the test is halted and the
30 pressure recorded. If no venting has been recorded by 14 kg/cm² the cap is recorded as having a venting pressure of 14+ kg/cm².

After the venting pressure test has been finished the plastic bottle top, with the cap attached, is removed and
35 is clamped in a Kork-a-Torque tester with which the

torque required to remove the cap from the bottle is measured.

The process of the invention can produce a standard gasket having a venting pressure of at least 10.5 kg/cm^2 (150 psi) and generally at least 12 kg/cm^2 . The preferred process can provide a gasket having a venting pressure of $14+ \text{ kg/cm}^2$ (200+ psi). The process can provide a gasket which has a removal torque of at least 6.5 cm kg (6 inch lbs) and generally at least 7.5 cm kg. 10 The removal torque preferably is less than 15.5 cm kg (14 inch lbs) and generally is less than 14, and preferably less than 13 cm kg. Best results are obtained when the removal torque is from 8 to 11 cm kg.

Preferably at least 60%, and generally at least 70%, 15 of the composition is provided by thermoplastic polymeric material, since it seems that the presence of large amounts of other organic materials, for instance large amounts of fusible tackifying resin as in Specification No. 1092161, prevent the attainment of the defined 20 physical properties. It is often preferred that the amount of thermoplastic polymeric material is 80 to 98%.

The thermoplastic polymeric material may be selected from any thermoplastic materials that have the specified viscosity when formulated with the slip aid and, if 25 present, the wax and/or the defined small amount of plasticising agent. The thermoplastic material may be selected from a wide range of thermoplastic polymers, for instance polyethylene, polypropylene, polybutylene, polystyrene, polyvinyl chloride, polyvinyl acetate, 30 copolymers of ethylene propylene or ethylene and styrene, copolymers of styrene with maleic anhydride, methacrylate, ethylacrylate or acrylonitrile, polymers and copolymers of conjugated dienes such as polybutadiene, polyisoprene, polychloroprene, styrene 35 butadiene, ethylene propylene diene rubber, acrylonitrile

styrene butadiene rubber, polyamides, polyester amides, polyurethanes, and cellulose esters such as cellulose acetate butyrate, cellulose acetate propionate and cellulose propionate. Mixtures may be used. The preferred polymers are one or more ethylene vinyl acetate copolymers, optionally blended with a polyalkylene, generally polyethylene.

The molecular weight of the thermoplastic material or materials and their chemical constitution must be selected such that the composition has the desired viscosity, and is not so low that the material should be defined as a wax.

It is often preferred that the thermoplastic material, for instance the ethylene vinyl acetate copolymers, or blend of such copolymers, should have a melt index of above 350 and generally above 450, most preferably from 500 to 600. This indicates a low molecular weight. It is then preferred for the amount of plasticising resin to be from 0 to 8%, preferably 0%. However good results can also be obtained with lower melt indices. For instance satisfactory compositions can be made when the melt index of the thermoplastic material is as low as 100, especially if the composition contains 15 to 30% wax and 0 to 8%, preferably 0%, plasticising resin. If the melt index of the thermoplastic is below 100, for instance 30 to 99, it will generally be desirable to include plasticising resin, for instance in an amount of 8 to 20%, often 12 to 18%.

As mentioned the composition may contain a wax or waxes, for example polyethylene wax, paraffin wax or microcrystalline wax. The amount is generally below 30%, for instance 15 to 27%. Suitable polyethylene wax, especially for blending with ethylene vinyl acetate copolymer, has a viscosity at 171°C and 237 sec⁻¹ shear rate of less than 50 poise, generally less than 40 poise

and typically between 0.5 and 20 poise. The polyethylene wax may be mixed with a higher molecular weight polyethylene which would generally have a melt index of greater than 100, typically 200-500. The amount of polyethylene in the blend with the ethylene vinyl acetate copolymer is generally 5 to 40%, preferably 10 to 30% and most preferably 12 to 20%.

The ethylene vinyl acetate in the blend may have quite a low vinyl acetate content in order to ensure that the two polymers are fully compatible in the composition. The amount of vinyl acetate may be below 30% and is often below 25%, for instance 10 to 19%.

Instead of producing a physical blend of polyethylene and ethylene vinyl acetate it is also possible to achieve the same results by forming a block copolymer of ethylene vinyl acetate and polyethylene.

Suitable plasticising resins that may be included in small amounts include hydroabietyl alcohol, terpene resin, glycerol ester of wood rosin-maleic anhydride adduct, hydrogenated, disproportionated or polymerised wood rosin, polyhydric alcohol esters of abietic acid or of hydrogenated wood rosin.

The polyhydric alcohol ester is usually a glycerol, ethylene glycol, diethylene glycol, or pentaerythritol ester. The pentaerythritol ester of hydrogenated wood rosin (often called merely "rosin" instead of "wood rosin") can for example be the stabilised form available as Hercules Resin pentalyn H. The polyhydric alcohol esters of abietic acid can conveniently be employed in the form of esterified wood rosin, since wood rosin contains a large proportion of abietic acid. Derivatives of wood rosin, i.e. hydrogenated, disproportionated or polymerised wood rosin, can also be used as the resin component. Preferred examples of the resin component are the glycerol ester of abietic acid, the ethylene

glycol ester of abietic acid, and the diethylene glycol ester of abietic acid, particularly the glycerol ester of abietic acid.

5 The molecular weight of the resin is generally from 300 to 3000. Often the total amount of plasticising resin is below 5%.

10 The slip aid may be a conventional silicone slip aid, a hydrogenated castor oil wax or a fatty amide, for instance an aliphatic amide in which the aliphatic group contains 10 to 30, preferably 14 to 25, carbon atoms, for instance being stearamide or oleamide. Preferably it is a blend of silicone oil and fatty amide, for instance in the proportions by weight 1:0.5 to 1:2.

15 The composition may include inorganic pigment or filler, as mentioned above, and may include other minor components for example antioxidants. The total amount of such other minor components is generally below 10%, preferably below 5%, by weight of the organic components of the composition.

20 The compositions are of particular value when the cap is of polypropylene or other plastic material but they are also useful for aluminium or other metal caps. The caps may be conventional screw-on caps or they may be roll on caps.

25 The gaskets are of particular value for sealing pressurised containers that are not heated, for instance for carbonated soft drinks. However the gaskets can also be used in non-pressurised containers and can be used in containers that are filled hot or pasteurised.
30 Naturally the temperature or other conditions of filling or pasteurisation must not be such as to damage the gasket.

The most important and valuable use of the compositions is for melt application to a plastic cap to
35 form a gasket for pressurised container, preferably for a

carbonated beverage, the cap preferably being a screw-on cap.

The following are examples of the invention.

EXAMPLE 1

5 85 parts of ethylene vinyl acetate copolymer having a melt index of 530 and containing 19% vinyl acetate is blended with 15 parts of polyethylene wax having a melt viscosity of 1.65 poise at 237 sec^{-1} shear rate and 171°C, 1 part of titanium dioxide, 1 part silicone slip
10 aid and 0.2 parts thermal stabiliser. The silicone slip aid is a 60,000 cS polysiloxane. The components are blended in a suitable mixer and are extruded, without addition of foaming gas, through a Nordson Foam Melt apparatus at a temperature of 175°C. The composition has
15 a viscosity of 152 poise at 171°C and 237 sec^{-1} shear rate. The molten composition is extruded into nominal 28 mm polypropylene screw caps which are spun at 6,000 rpm. The deposited film weight, per cap, is 402 mg. Upon testing, as above, venting pressures between 2.1 and
20 5.3 kg/cm^2 and removal torques of 16.5 and 12 cm kg are recorded. Accordingly this composition is unsatisfactory.

When the composition is reformulated using 2 parts of the slip aid, instead of 1, venting pressures of 10.5,
25 14+ and 14+ kg/cm^2 and removal torques of 12.1, 13.2 and 14.3 cm kg are recorded. Accordingly this composition is satisfactory.

EXAMPLE 2

The process of Example 1 is repeated except that the
30 amount of ethylene vinyl acetate is reduced to 80 parts and the amount of polyethylene wax is increased to 15.9 parts and 4.05 parts of polyethylene with a melt index of 250 is included. The viscosity at 171°C and 237 sec^{-1} is 159 poise. The film weight is 411 mg. Venting
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pressures of 14+ and 14 kg/cm² and removal torques of 15.4 and 13.2 cm kg are recorded.

When this is repeated using 2 parts silicone slip aid, instead of 1 part and a film weight of 365 mg
5 venting pressures of 14+ kg/cm² are consistently recorded and the removal torque varies between 13.2 and 14.3 cm kg.

When the process is repeated using 1 part silicone slip aid and 0.6 parts oleamide slip aid and a coating
10 weight of 354 mg, the venting pressure is consistently recorded as 14+ kg/cm² and the removal torque as 7.8 cm kg.

EXAMPLE 3

A process is conducted as in Example 1 except that a
15 composition is prepared from 70 parts ethylene vinyl acetate, 30 parts polyethylene wax, 1 part oleamide and 1 part thermal stabiliser and is deposited to form a gasket of 341 mg. The venting pressure is 14+ kg/cm² and the removal torque 7.8 cm kg.

20 EXAMPLE 4

A process is conducted as in Example 1 except that the composition is formed of 74 parts of an ethylene vinyl acetate copolymer blend having a melt flow index of 126.5 and formed of equal parts of copolymers of melt
25 index 40 and 400, 26 parts microcrystalline wax, 1 part titanium dioxide, 1 part silicone oil, 1 part fatty amide and 0.3 parts thermal stabiliser. The viscosity of the composition at 171°C and 237 sec⁻¹ is 150 poise. Upon lining as in Example 1 the film weight is 262 mg, the
30 venting pressure 14 + kg/cm² and removal torque 139 cm kg.

EXAMPLE 5

A process is conducted as in Example 1 except that the composition is formed from 74 parts of an ethylene
35 vinyl acetate copolymer blend having a melt flow index of

126.5 and formed of 21 parts copolymer melt index 7 and
53 parts copolymer melt index 400, 26 parts
microcrystalline wax, 1 part titanium dioxide, 1 part
silicone oil, 1 part fatty amide and 0.2 parts thermal
5 stabiliser. The viscosity of the composition at 171°C
and 237 sec⁻¹ is 155 poise . Upon lining as in Example
1 the film weight is 237 mg, the venting pressure
14 + kg/cm² and removal torque 12.7 cm kg.

EXAMPLE 6

10 A process is conducted as in Example 1 except that
the composition is formed of 60 parts ethylene vinyl
acetate copolymer having a melt index 40, 5 parts
petroleum jelly (Vaseline, trade mark), 15 parts
pentaerythritol rosin ester plasticising resin, 5 parts
15 microcrystalline wax, 15 parts high melting point
paraffin wax, 1 part titanium dioxide, and 0.5 parts
thermal stabiliser. The viscosity at 171°C and 237 sec⁻¹
is 153 poise Upon lining as in Example 1 the film weight
is 253 mg, the venting pressure 14 + kg/cm² and removal
20 torque 16.0 cm kg.

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CLAIMS

1. A method of forming a sealing gasket on the inner surface of a screw closure for a container by applying to the surface an unfoamed molten composition comprising
5 thermoplastic material characterised in that the composition comprises 55 to 99% by weight thermoplastic polymeric material, 0 to 30% of a wax, 0 to 20% plasticising resin and 0.1 to 10% slip aid (all percentages being by weight based on the weight of
10 fusible organic components in the composition) and the composition has a viscosity at 172°C measured at a shear rate of 237 sec⁻¹ of from 50 to 400 poise and that when applied as a melt to a closure to form a standard gasket, as herein defined, provides a gasket that has a venting
15 pressure, as herein defined, of at least 10.5 kg/cm² and that has a removal torque, as herein defined, of from 6.5 to 19.5 cm kg.
2. A method according to claim 1 in which the amount of thermoplastic polymeric material is from 70 to 99% and
20 the amount of plasticising resin is 0 to 8%.
3. A method according to claim 1 or claim 2 in which the composition has a viscosity of from 110 to 180 poise.
4. A method according to any preceding claim in which the thermoplastic polymeric material has a melt index of
25 above 350.
5. A method according to any of claims 1 to 3 in which the thermoplastic polymeric material has a melt index of above 100 and the composition contains from 15 to 30% wax and 0 to 8% plasticising resin.
- 30 6. A process according to any one of claims 1 to 3 in which the thermoplastic material has a melt index of less than 100 and the composition contains from 8 to 20% plasticising resin.
7. A process according to any one of claims 1 to 5 in
35 which the composition contains 0% plasticising resin.

8. A process according to any preceding claim in which the thermoplastic polymeric material comprises ethylene vinyl acetate copolymer.
9. A process according to any preceding claim in which
5 the amount of thermoplastic polymeric material is from 60 to 85% and the amount of wax is from 15 to 27%.
10. A process according to any preceding claim in which the amount of slip aid is 1 to 5%.
11. A process according to any preceding claim in which
10 the slip aid comprises a silicone, a fatty amide or a blend thereof.
12. A process according to any preceding claim in which the gasket has a removal torque of 7.5 to 14 cm kg and a venting pressure of at least 12 kg/cm².
- 15 13. A process according to any preceding claim in which the screw closure is a plastic screw-on cap or a plastic roll-on cap.
14. A screw on closure, or a pressurised container sealed by a screw-on closure in which the closure is a plastic
20 screw-on cap or plastic roll-on cap and the closure carries a gasket characterized in that the gasket has been formed by a process according to any of claims 1 to 13.

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DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (Int. Cl. ³)
X	DE-A-1 544 989 (W.R. GRACE & CO.) * the whole document *	1-14	B 65 D 53/00
X	FR-A-2 442 777 (JAPAN CROWN CORK) * claims 1-5 *	1-14	
X	DE-A-2 932 200 (VEREINIGTE METALLWERKE RANSHOFEN-BERNDORF AG) * page 3, lines 31-34 *	7	
			TECHNICAL FIELDS SEARCHED (Int. Cl. ³)
			B 65 D C 08 L
The present search report has been drawn up for all claims			
Place of search THE HAGUE		Date of completion of the search 11-09-1984	Examiner GOOVAERTS R.E.
CATEGORY OF CITED DOCUMENTS		T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document	
X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document			