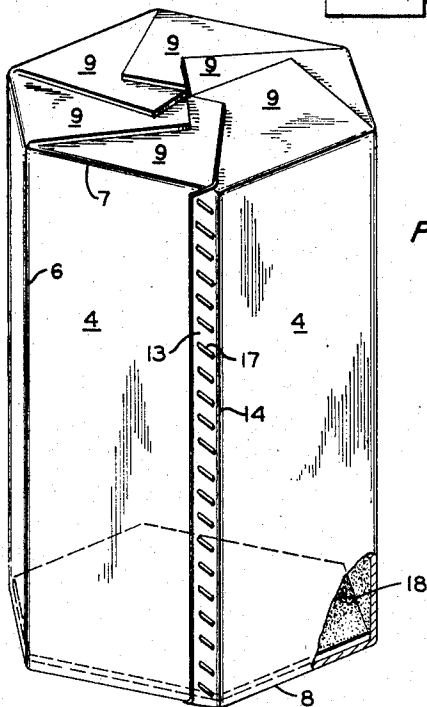
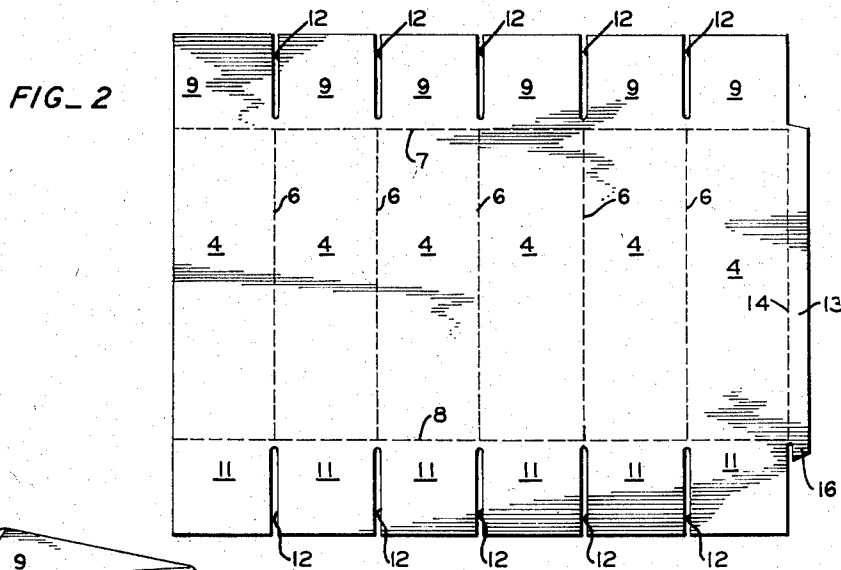
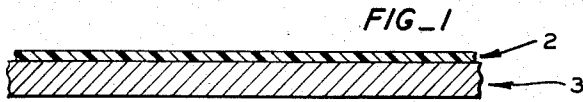


Jan. 20, 1959

M. P. MARANDER ET AL
PAPER PRODUCTS HAVING A RELEASE COATING
AND METHOD OF PREPARATION THEREOF
Filed Oct. 14, 1957

2,869,722



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2,869,722

PAPER PRODUCTS HAVING A RELEASE COATING AND METHOD OF PREPARATION THEREOF

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Application October 14, 1957, Serial No. 689,805

7 Claims. (Cl. 206—84)

This invention relates to paper products that have a release coating to prevent adherence of materials with tacky or adhesive characteristics, and most particularly to paperboard containers having a coating that readily releases packaged tacky materials such as asphalt and crude rubber.

The use of paperboard containers for shipping tacky or adhesive substances has long presented a problem because of the tendency of such materials to adhere to the paperboard. Although many types of coatings have been employed on paper products in order to prevent tacky materials from adhering to the paper, none of such coatings have been satisfactory for all purposes. The packaging of asphalt in paperboard containers has presented a particular problem since molten asphalt is poured into the containers at elevated temperatures as high as 450° F. When the molten asphalt sets, it forms a solid mass which tends to adhere to the containers.

One type of coating heretofore employed is formed primarily of a loosely bound clay strip coating which is designed to provide a barrier that prevents the tacky material from contacting and adhering to the paperboard. However, when molten asphalt is poured into a clay coated container at elevated temperatures, the asphalt often penetrates the clay and sticks to the fibers of the paperboard container. This problem is greatly increased when the container is handled roughly before it is filled, since the clay coating is loosely bound and readily flakes off the interior of the empty container thereby leaving voids in the coating. Furthermore, when the container is separated from the asphalt, the clay tends to adhere to the asphalt and contaminate it.

Another conventional type of strip coating for paperboard containers is formed by providing paperboard, with two layers of different coatings. The exposed or top coating prevents penetration of the asphalt, and the undercoating is loosely bound for easy cleavage. When the asphalt or other tacky packaged material is removed from such paperboard containers, the exposed coating on the paperboard tends to adhere to the packaged material and the undercoating cleaves adhering partly to the paperboard and partly to the top coating and the asphalt. However, this type of strip coating also prevents a contamination problem, and has the additional disadvantage of requiring the application of two coats.

The problems encountered with the strip coatings previously employed for packaging tacky materials in paperboard containers have been overcome by the present invention in which a single release coating is provided which forms a barrier that prevents contact of asphalt or tacky materials with the paperboard, even under adverse filling conditions. Asphalt or other tacky material is released at the surface of the coating so that the coating remains essentially completely on the paperboard and does not adhere to the packaged tacky material.

Summarizing this invention, a release coating having the foregoing desirable features is provided by mixing

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a film-forming polymeric carrier material and a heat curable silicone resin, applying the resultant mixture together with a catalyst to the paper product, and curing the mixture to provide a true release coating and a barrier to penetration by tacky materials. The combination of the film-forming polymeric material with a heat curable silicone resin provides a synergistic effect for coating a porous product, particularly cellulosic materials such as paper and paperboard, and the mixture shows far superior properties as a release coating than equivalent amounts of either of its components employed alone.

In a coating formed from the mixture of film-forming carrier and silicone resin, the polymeric film-forming carrier acts as a barrier to prevent penetration of asphalt or other tacky material into the paperboard, and also serves as a carrier or bodying agent for the silicone resin. As a result, the silicone resin is held at the surface of the paperboard where its release properties are most effective. If the film-forming carrier is not included in the mixture, the silicone resin tends to be absorbed into the porous paperboard, and relatively large amounts of the expensive silicone resin are required to produce a satisfactory release coating. Consequently, it is apparent that the present invention is particularly valuable for providing a release coating on porous surfaces, such as in paperboard containers. The coating hereof enables tacky materials, such as asphalt, crumb rubber, surgical tape and masking tape to be released readily from paper or paperboard, and thus paperboard containers can be used to package such tacky materials.

In the drawings:

Fig. 1 is an enlarged fragmentary sectional view of paperboard coated in accordance with this invention, which for purposes of clarity is more or less schematic in illustrating the relative thicknesses of the various layers.

Fig. 2 is a plan view of a typical paperboard container blank employed to form an asphalt container.

Fig. 3 is a perspective view of an assembled asphalt container viewed from above.

The coating of silicone resin and film-forming carrier 2 is applied to paperboard 3 on the side of the paperboard which contacts the tacky material. When the silicone resin-polymeric carrier coating is used on corrugated paperboard, it has been found convenient to apply the coating to the paper web before the corrugated paperboard is formed. Any conventional means, such as the usual roll coating method may be utilized to apply the release coating while it is in the form of a liquid. A catalyst for polymerizing the silicone resin is added to the liquid release coating mixture before the release coating is applied, and the coated paper is subjected to elevated temperatures above about 200° F. to cure the coating after it has been applied.

Figs. 2 and 3 illustrate an example of a typical paperboard container for asphalt, and the release coating hereof is advantageously employed on such paperboard asphalt containers. Containers of this type are described in greater detail in United States Patent No. 2,761,611. The blank shown in Fig. 2 is divided into six rectangular body panels 4 by five longitudinal crease lines 5. A transverse top crease line 7 spaced downward from the top of the blank, and a transverse bottom crease line 8 spaced upward from the bottom of the blank define material on the blank above and below the top and bottom crease lines which forms the top and bottom end flaps 9 and 11. Cuts 12 are formed in line with the longitudinal crease lines 5 and extend inwardly from the top and bottom edges of the blank terminating shortly before reaching the top and bottom transverse crease lines.

Along one outside edge of the blank is a stitching flap 13 of a width substantially less than the width of body panels 4 and separated from the adjacent body panel by a longitudinal crease line 14. A tab 16 extending from flap 13 is stitched to a bottom end flap 11 in the set up carton.

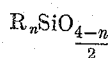
In order to form the blank into the container shown in Fig. 3, the stitching flap 13 is brought around on top of the outside marginal edge of the body panel 4 at the opposite side of the blanks. Metallic staples 17 are conveniently employed to stitch the stitching flap 13 to body panel 4, and to stitch tab 16 to the bottom end flap 11.

Usually, the containers are stitched in this manner and then shipped while collapsed to the place where they are to be filled. The collapsed container is erected by the workman, the bottom flaps 11 are closed and the molten asphalt 18 is poured into the container at temperatures as high as 450° F. Bottom pads, not shown, are often used to prevent the molten asphalt from leaking, and such pads also are advantageously coated with the release coating hereof. Finally, the top flaps 9 are closed and the asphalt is allowed to cool.

After the asphalt has solidified, the package is shipped to the user, who generally slits the paperboard container with a sharp instrument and peels the paperboard from the asphalt. The coating hereof enables the paperboard to be easily and cleanly separated from the asphalt.

Silicone resins having release characteristics are well known and are available commercially. Any of such heat curable silicone resins that are solvent soluble, water dispersible, and which can be catalytically polymerized may be employed with the film-forming carrier to provide the release coating for paper products. Such resins are usually either organo polysiloxane resins with siloxane linkages in which the silicon atoms are interconnected through oxygen atoms, organo polysilane resins in which the silicon atoms are directly linked in the polymer chain, or silicone resins containing both siloxane and silane linkages. These silicone resins are employed in the coating composition hereof when they have been reacted to the point at which they are still soluble in organic solvents, such as toluene and benzene, and are dispersible in water. They can be readily further polymerized and cured to form an infusible, insoluble cross-linked polymer when they are mixed with a catalyst and subjected to heat.

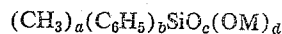
The polysiloxane resins that have been particularly useful for forming the release coating hereof have a repeating unit characterized by the formula



In this formula the organic radical R is selected from the group consisting of alkyl, aryl, alkaryl, aralkyl, alkenyl, and combinations of such groups. Best results are obtained with the simpler organic groups such as methyl, ethyl, phenyl or hydrogen. Si is silicon, O is oxygen and n is equal to between about 1.0 and 2.0. Examples of suitable organo polysiloxane resins are disclosed in U. S. Patent Nos. 2,610,169, 2,482,276 and 2,491,843, as well as in numerous other patents. These resins are solvent soluble, water dispersible polymers which form cross-linked infusible polymers when further polymerized, and they are advantageously combined with a film-forming carrier to form a release coating for paperboard.

Another silicone resin that may be employed in the composition hereof is a polymer that contains both polysilane and siloxane type bondings. In these resins some of the silicon atoms are interconnected by silicon-to-silicon bonding, and some of the silicon atoms are interconnected through intermediate oxygen bonding. For example, U. S. Patent No. 2,672,104 describes such resins that can be used in making paperboard release

coatings hereof as heat curable silicone resins having an average composition represented by



where M is selected from the group consisting of hydrogen atoms and primary and secondary alkyl radicals; $a+b$ has a value of from 1.4 to 2, c has a positive value, d has a value of 0.01 to 1, and $a+b+2c+d$ has a value of up to 3.5. Between 30 and 75 percent of the total phenyl and methyl radicals are phenyl radicals. Such resins are solvent soluble and water dispersible polymers which can be further cured and cross-linked to form an infusible, insoluble polymer which has release characteristics.

In addition well known solvent soluble, water dispersible, and heat curable organo polysilane resins which have release properties may be employed as the silicone resin portion of the release coating. For example, suitable polysilane resins in which the silicon atoms are connected to adjacent silicon atoms in the polymeric chain are disclosed in United States Patent 2,563,005.

In order to employ the organo silicone resins as a coating in combination with the water soluble film-forming polymeric carrier, such resins are most advantageously utilized in the form of dispersions of the resins in water. However, solutions of the silicone resins in solvents, such as toluene, xylene, and benzene, may also be employed.

Suitable dispersions of the resins are well known and are available commercially. Also, the methods of preparing such dispersions of solvent soluble polymeric resins are well established. The silicone resins are dispersed in water by adding any of the surface active agents used for such purposes to a liquid mixture of water and the silicone resin, and then mechanically agitating the mixture to form the dispersion. The surface active or in other words emulsifying agents may be either cationic, anionic or nonionic, and they are readily available commercially. Examples of suitable surface active agents are the morpholine salts of fatty acids wherein the fatty acid contains from 12 to 18 carbon atoms, such as morpholine oleate and morpholine stearate. Other suitable emulsifying agents including acetates of n -primary amines having from 12 to 18 carbon atoms in the amine chain such as n -dodecyl amine acetate, the sodium salt of alkylated arylpolyether sulfates, and fatty acids of 12 to 18 carbon atoms such as hexaethylene glycol monooleate. United States Patent Nos. 2,666,685 and 2,586,211 disclose methods of preparing emulsions of silicone resins.

Emulsions of organo silicone resins that may be used in the coating hereof and which are available commercially include Dow Corning Corporation emulsions XE-5067 and Dow Corning 22. These resin emulsions, supplied at 40 percent total solids by weight are basically methyl silicone resins which can be catalytically cross-linked. Dow Corning Corporation catalyst XEY-21, which is a 20 percent emulsion of mixtures of metal organic salts, is useful as a catalyst for such emulsions. Union Carbide and Carbon Emulsions Y-1006, Y-1383 and Y-1384 are also advantageously employed in the release coating hereof. These emulsions contain 30 percent silicon solids by weight, they contain a silanic hydrogen containing copolymer, and are advantageously catalyzed by zinc octoate. Similarly, General Electric sells 81814 Silicon Emulsion which is available at 50 percent total solids content and which may be employed in the release coating hereof. This emulsion contains a curable methyl silicone, and a zirconium acetate catalyst is recommended for cross-linking the resin.

Film-forming carriers that are mixed with the organo silicone resin solutions or aqueous dispersions to provide the release coating are natural or synthetic polymers which are soluble in either aqueous or organic solvents and are sufficiently compatible with the organo silicone polymers to enable the two components to be used as a mix-

ture. The film-forming carriers in the form of liquid solutions are mixed with the silicone resin and the mixture is applied to the paperboard while the film-forming carrier is in the form of a liquid solution. When the release coating is to be employed on paperboard asphalt containers, oil and heat resistant water soluble film-forming polymeric carriers are utilized in aqueous solution. For example, methyl cellulose, polyvinyl alcohol, hydroxyethyl cellulose and sodium carboxymethyl cellulose are particularly valuable in the composition hereof and produce superior release coatings when employed with the silicone resin. Other suitable film-forming carriers include casein and other water soluble proteins, starches, modified starches, sodium alginates and sodium acrylates. Where heat resistance is not required but properties such as water resistance are desirable, water insoluble polymeric carriers may be used in which case it is convenient to employ the mixture dissolved in a solvent. Suitable solutions of film-forming carriers include vinyl chloride dissolved in ketone solvents such as methyl ethyl ketone, styrene dissolved in aromatic solvents such as benzene, xylene or toluene, and butadiene-styrene or butadiene-acrylonitrile copolymers dissolved in aromatic solvents.

Particularly excellent results are obtained with polyvinyl alcohol film-forming carriers. The insoluble polyvinyl alcohol compositions are available in the form of aqueous emulsions, and solutions of polyvinyl alcohol can be prepared by application of heat. When the insoluble polyvinyl alcohol is incorporated in the final release coating composition, the final dry release coating film has an increased resistance to moisture because of the low solubility of the polyvinyl alcohol in water at ordinary temperatures. As a result such release coatings including the polyvinyl alcohol retain their release properties even under damp or moist conditions.

None of these film-forming carriers provide release coatings for tacky materials such as asphalt, when they are used alone without the organo silicone resin. The aqueous mixture of organo silicone resin and film-forming polymeric carrier is stable and can be stored for appreciable periods before it is applied to paperboard.

In addition to the silicone resin and the film-forming polymeric carrier, the coating may include conventional additives for polymeric coatings such as any dyes, pigments, and plasticizers that may be desired for a particular application. However, for most purposes such other additives need not be employed.

The proportions of organo silicone resin to film-forming carrier may vary considerably, and the coating will still provide excellent release characteristics. The ratio of silicone resin to film-forming polymeric carrier may range between about 5/95 and 95/5. In other words the amount of silicone resins may vary between about 5 percent and 95 percent by weight, and the amount of film-forming polymeric material may range from about 5 to about 95 percent based upon the total weight of silicone resin and film-forming carrier solids in the mixture. If more than about 95 percent of silicone resin is included in the mixture, an unduly large amount of the coating must be applied to provide the desired release characteristics. This is due to the decrease in effectiveness of the film-forming carrier in holding the silicone on the surface of the paper when less than 5 percent of carrier is present. As a result the coating becomes extremely expensive to employ in large scale paperboard applications if only small amounts of film-forming polymer are included in the coating. Also, if less than about 5 percent of silicone is employed in the coating, the release characteristics of the coating are considerably diminished. The film-forming carrier in itself does not possess the requisite release characteristics, and it is useful only when combined with more than about 5 percent of the silicone resin. Extensive tests have demonstrated that the best economical release coatings are obtained

with a ratio of silicone resin to film-forming polymer of between 20/80 and 50/50 based on the weight of silicone resin and film-forming polymeric carrier solids present in the coating.

Shortly before the aqueous mixture of silicone resin emulsion and film-forming polymeric carrier is applied to the paperboard, sufficient catalyst for the silicone resin is added to polymerize the resin. Suitable catalysts for polymerizing the organo silicone resins hereof are well known. Examples of such catalysts are lead naphthenate, cobalt naphthenate, lead linoleate and zinc octoate. United States Patent Nos. 2,449,572; 2,610,169; 2,647,881 and 2,676,948 disclose catalysts for the coating compositions hereof. Commercial suppliers of such catalysts recommend the optimum amount of catalyst to be employed. The ratio of silicone resin catalyst to silicone resin varies between 1/99 and 20/80 on the basis of solids present, or in other words the amount of catalyst is between one percent and 20 percent of the amount of silicone resin in said binder. However, the amount of catalyst is not critical and depends upon the particular catalyst employed.

After the catalyst has been added, the mixture is stirred until homogeneous and then it is applied to the paper product in any conventional manner. The usual roll coating method is the most convenient way of applying the release coating to paperboard. In order to form a surface release coating on the paperboard the solids content and viscosity of the coating are adjusted depending upon coating methods to be employed and the porosity of the particular paperboard that is being coated. Generally for commercial roll coating equipment the ratio of solids to water or solvent is adjusted to provide a viscosity between about 500 to 10,000 centipoises.

The amount of coating applied per thousand square feet of paper product is particularly dependent on the type and porosity of the paper or paperboard. Usually between about 0.5 and 1.5 pounds of coating based upon the dry weight of silicone resin and film-forming carrier is applied per one thousand square feet of paper product, such as a paperboard container wall. On relatively impervious surfaces, much thinner coatings may be satisfactory.

After the mixture of silicone resin, film-forming carrier and catalyst have been applied to the paperboard, the resin is cured at elevated temperatures. For practical purposes it is generally desirable to heat the paperboard to a temperature of about 250° F. or above until the coating is cured. However, temperatures as low as 200° F. have proven to be satisfactory. It has been found that substantially completely cured coatings have the best release characteristics, and that the coatings are generally satisfactorily cured in between about 15 seconds to 4 minutes at the temperatures specified.

Extensive tests with various coatings applied to paperboard in accordance with this invention have demonstrated the superior properties of the coated paperboard hereof in releasing numerous tacky materials, including asphalt, crumb rubber, surgical tape and masking tape. Optimum release characteristics were obtained for almost all uses when the amount of organo silicone resin was 35 percent by weight and the film-forming polymer about 65 percent by weight based on the silicone resin and film-forming polymer solids in the coating. The preferred coatings provide such effective release characteristics that several times as much unmodified silicone emulsion is required to produce equivalent release of tacky materials from paperboard. In other words the amount of unmodified silicone resin required to produce satisfactory release of tacky materials from paperboard must be more than five times the amount of silicone resin in the preferred coating hereof which produces equivalent release.

The following are specific examples of the preparation of some of the coatings hereof and the application of such coatings to paper products:

Example 1

A coating composition was prepared containing about 65 percent methyl cellulose, 34 percent catalyzed silicone resin and 1 percent red dye in percent by weight solids. First, the methyl cellulose powder (25 centipoise grade) was stirred into hot water at a temperature of about 185° F. to provide a concentration of about 30 percent solids, and then diluted with cold water to a concentration of about 7.2 percent solids. This mixture of methyl cellulose and water was stirred until a uniform solution was formed, at which time the solution had a temperature of approximately 100° F.

Next an aqueous silicone resin dispersion of heat curable methyl siloxane resin containing about 40 percent by weight resin solids, 5 percent fatty alcohol amine sulfate dispersing agent, and an aqueous solution of the red dye was added to the methyl cellulose solution and the mixture stirred until homogeneous. The red dye serves as a guide to determine whether or not the film applied to paperboard is continuous. The solution was then stored.

Just prior to application of the coating to paperboard, sufficient lead naphthalene catalyst was added to provide 10 percent by weight catalyst based on the weight of silicone solids. This mixture was then stirred until homogeneous and contained about 10 percent by weight solids. The viscosity of the solution was about 2000 centipoises when it was applied to paperboard in a conventional roll coating apparatus. About 10 pounds of the coating composition was applied per 1000 square feet based on the wet weight of the coating.

After the coating had been applied, the coated paperboard was dried at a temperature of 300° F. in a tunnel dryer to remove water and cure the silicone resin. The cured coated paperboard was used to form a container for asphalt.

The asphalt container was set up and molten asphalt was then poured into the container. After the asphalt had solidified and cooled, the coated paperboard readily released from the asphalt.

Example 2

A release coating was prepared which contained 80 percent by weight polyvinyl alcohol powder and 20 percent by weight catalyzed silicone emulsion solids. First the polyvinyl alcohol was dispersed in cold water, and the dispersion was then heated to approximately 200° F. until the polyvinyl alcohol had dissolved. The final solution, which had about 10 percent solids, was cooled to about 90° F. A silicone aqueous dispersion containing 40 percent by weight silicone resin solids and 3 percent by weight morpholine oleate, in which the silicone resin was methyl phenyl polysilane having a ratio of organic radicals to silicone of about 1.5 in which about 50 percent of the organic radicals are phenyl was added with stirring, and the mixture was stored.

Shortly before the coating was to be applied to the paperboard, 2 percent by weight of zinc octoate catalyst based on the weight of silicone resin solids was mixed with the mixture of silicone emulsion and polyvinyl alcohol, and the mixture was applied to paperboard by the conventional roll coating method. The amount of coating applied to paperboard was about one pound (dry weight) per 1000 square feet.

After the paperboard had been coated, it was cured by subjecting it to a temperature of about 250° F. for a period of 3 minutes. The resultant coated paperboard released readily for molten asphalt, surgical tapes and crumb rubber.

Example 3

A release coating which is to be deposited on paperboard from an organic solvent solution was prepared by blending 25 parts of a 15 percent solids solution of methyl siloxane resin in xylene with 75 parts of a 15 percent solids solution of about 85 percent vinyl chloride—15 percent vinyl acetate copolymer resin in a 50/50 mixture of toluene and methyl ethyl ketone.

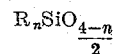
Just prior to application of the coating on paperboard, 6 percent zinc octoate on silicone solids was added as a catalyst. The catalyzed coating was applied on the paperboard on a roll coating apparatus at a rate of one pound per 1000 square feet (dry weight) and was heat cured at 275° F. for two to three minutes. The coatings were found to readily release a commonly used pressure-sensitive tape.

We claim:

1. A cellulosic container wall having a coating thereon comprising a mixture of a catalytically polymerized silicone resin with release properties, and a polyvinyl alcohol polymeric binder.

2. A paper product container wall having a coating thereon comprising a mixture of from about 5 to 95 percent by weight of a catalytically polymerized, heat-cured silicone resin with release properties; and from about 5 to 95 percent by weight of polymeric polyvinyl alcohol carrier based on the weight of silicone resin and carrier in said coating.

3. A paperboard container blank having a coating thereon comprising from about 5 to 95 percent by weight of a catalytically polymerized heat-cured silicone resin with repeating groups characterized by



in which R is selected from the group consisting of alkyl, aryl, alkaryl, aralkyl, and combinations of such organic radicals, Si is silicon, O is oxygen and n is between about 1.0 and 2.0; from about 5 to 95 percent by weight of polymeric polyvinyl alcohol carrier based on the weight of silicone resin and carrier in said coating; and a catalyst for said silicone resin present in an amount of between 1 percent and 20 percent of the amount of silicone resin in said carrier.

4. Paperboard having a coating thereon comprising from about 5 to 95 percent by weight of a catalytically polymerized, heat-cured silicone resin that has release properties; and from about 5 to 95 percent by weight of polymeric polyvinyl alcohol carrier based on the weight of silicone resin and carrier in said coating, said coating being present on said paperboard in an amount of from 0.5 to 1.5 pounds per 1,000 square feet.

5. A package comprising a paperboard container having a coating on its interior walls of from about 5 to 95 percent by weight of a catalytically polymerized heat-cured silicone resin with release properties, from about 5 to 95 percent by weight of polymeric polyvinyl alcohol carrier, said percents by weight being based upon the weight of silicone resin and carrier in said coating, and a catalyst for said silicone resin present in an amount of between 1 percent and 20 percent of the amount of silicone resin in said coating; and a tacky material within said container.

6. The method of preparing a paperboard container that releases readily from tacky materials which comprises forming a mixture of from about 5 to 95 percent by weight of a silicone resin and from about 5 to 95 percent by weight of a solution of polyvinyl alcohol, said percents by weight being based on the weight of silicone resin solids and polyvinyl alcohol solids in said coating; adding to said mixture a catalyst for said silicone resin, applying said mixture to paperboard, and heating the coated paperboard at a temperature of over 200° F. for a period of over 15 seconds to cure the silicone resin and form a release coating.

7. The method of preparing a paperboard container that releases readily from tacky materials which comprises forming a mixture of from about 5 to 95 percent by weight of an aqueous dispersion of a silicone resin and from about 5 to 95 percent by weight of an aqueous solution of polyvinyl alcohol, said percents by weight being based on the weight of the silicone resin solids and polyvinyl alcohol solids in said coating; adding to said mixture from between about 1 to 20 percent by weight of a catalyst for said silicone resin based on the weight of silicone resin in said mixture, applying said mixture to paperboard in an amount of between about 0.5 and 1.5 pounds per 1,000 square feet based on the

weight of solids in said mixture, and heating the coated paperboard at a temperature of over 200° F. for a period of over 15 seconds to cure the silicone resin and form a release coating.

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UNITED STATES PATENT OFFICE
CERTIFICATE OF CORRECTION

Patent No. 2,869,722

January 20, 1959

Myron P. Marander et al.

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction and that the said Letters Patent should read as corrected below.

Column 1, line 57, for "prevents" read -- presents --; column 7, line 29, for "naphthalene" read -- naphthenate --; line 60, for "silicone" read -- silicon --.

Signed and sealed this 2nd day of June 1959.

(SEAL)

Attest:

KARL H. AXLINE

Attesting Officer

ROBERT C. WATSON
Commissioner of Patents