[Second Edition.]

PATENT SPECIFICATION

437,304

Convention Dates (United States)

Jan. 20, 1933:
Dec. 22, 1933:

Corresponding Applications in United Kingdom

No. 2159/34.
No. 2160/34.

Dated Jan. 22, 1934

(One Complete Specification Left under Section 91 (2) of the Patents and Designs Acts, 1907 to 1932.)

Specification Accepted: Oct. 22, 1935.

COMPLETE SPECIFICATION.

Improvements in and relating to Thermoplastic Rubber Derivatives.

We, WINGFOOT CORPORATION, a corporation organized and existing under the laws of the State of Delaware, United States of America, located at 1144, East Market Street, Akron, State of Ohio, United States of America, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to thermoplastic rubber derivatives. It includes compositions containing them and products made from them. Among the products are coating compositions, coated objects and a sheet or film. The preferred form of sheet or film is a transparent wrapping material which is moistureproof even though no thicker than one-thousandth of an inch and which may be sealed merely by the application of heat and pressure. It may be made of a halogen-containing rubber derivative, such as rubber hydrochloride which is stabilized, either by special processing or by admixture with suitable materials, and remains flexible even on prolonged exposure to light.

Rubber is an unsaturated hydrocarbon and as such is capable of reacting additively with various substances which are thought, for the reason that the addition of such substances has been found to decrease the degree of chemical unsaturation of the rubber, to attach themselves at the double bonds of the rubber molecule. Natural rubber, for example such as that from the tree Hevea Brasiliensis has been determined to have the empirical formula $C_5H_8)_x$ in which the value of x has not as yet been ascertained but in which there is known to be one double bond for each C_5H_8 group.

When rubber is saturated with hydrogen chloride the empirical formula of the material is apparently changed from $(C_5H_8)_x$ to $(C_5H_9Cl)_x$. This product as made heretofore has not the resiliency, flexibility and tear resistance of rubber but is brittle. If only partially saturated with hydrogen chloride so that the product contains about 28% to 30.5% of chlorine a flexible product is obtained, which in this respect at least is markedly different from derivatives made by completely saturating rubber with hydrogen chloride according to the processes of the former art.

Various chlorinated rubbers may be produced. The double bonds may be saturated with chlorine to form a compound with the empirical formula $(C_5H_8Cl_2)_x$ and by substitution chlorinated compounds of varying chlorine content may be obtained. The chlorine may be both substituted and added in the same compound.

Rubber also reacts with other halides and hydrohalides to form related compounds such as rubber hydrobromide and brominated rubbers. The rubber may be both halogenated and hydrohalogenated as a chlorinated rubber hydrochloride. The same rubber compound may contain different halogens combined with the C_5H_8 nucleus, as for example, chlorine and iodine or bromine.

Although various such compounds are described in the literature, until this invention no one had formed a satisfactory wrapping material from any such compound. Although it was known that chlorinated rubber, for example, could be formed into a transparent film, the film produced was not a wrapping material because it did not remain flexible but quickly became brittle on exposure to light. A wrapping material must be flexible and must remain flexible during a reasonable period of use even though exposed to sunlight. Materials which quickly become brittle in sunlight, although they may be used for wrapping quickly perishable foodstuffs, such as bread, are not generally satisfactory for wrapping and are not considered wrapping materials in the sense in which that term is used herein.

A wrapping material, as that term is used in the appended claims means a flexible covering material which has sufficient strength to withstand wrapping operations, either manual or mechanical, and to withstand ordinary handling. It must be of such a composition that it will not contaminate the material wrapped. It must be sufficiently stable so that it will not deteriorate rapidly, but will retain its covering properties during storage and subsequent handling.

According to the invention a composition of desirable stability comprises a halogen containing rubber derivative which contains one or more of the following compounds:

Hexamethylene tetramine
Dicyclohexylamine
Ditetrahydrofurfurylamine
Methyleneaminoacetonitrile
Cyclohexylamine-formaldehyde
Cyclohexylaminoacetonitrile
Tetramethyl thiuram disulphide
Diphenylguanidine
Dihydroxy diphenyl propane,

which compounds are referred to herein as photochemical inhibitors.

In its preferred form the composition of matter of the invention is a film which may be 0.001 inch thick and serves as a wrapping material, which retains its flexibility for a relatively long period. Rubber hydrochlorides containing more than about 30.5% chlorine or less than about 28 or 29% chlorine are not so suited for the manufacture of wrapping material. Rubber hydrochlorides which contain at least 29—30.5% of chlorine are, when formed into a thin film, as described below, clear, substantially colorless, transparent, flexible, extensible to a slight degree and highly resistant to tear. Such a film has a low flammability, burning very slowly with a low flame. It is resistant to water, moderately concentrated acids and alkalis, mineral and vegetable oils and most organic solvents. It is not tacky at ordinary temperatures and wholly different from the rubber from which it is prepared. It softens somewhat and becomes sticky at temperatures in excess of 110° C., a property which is valuable in that it permits films to be spliced together and packages to be sealed by means of a hot roller or other similar application of heat and pressure. Splicing may also be effected by the use of solvents, such as benzene, or chloroform or a cement formed by dissolving rubber hydrochloride in such as solvent. The films may be coloured by soluble dyes, such as oil soluble dyes if desired, and also by insoluble coloring ingredients if a clear transparent film is not required. Also they may be printed with inks ordinarily used for this type of work. They may be embossed to give pleasing surface finishes.

Certain of the compounds mentioned herein prolong the life of the film in which they are included as much as ten or fifteen times or longer, and a rubber hydrochloride film containing 29.5% chlorine and a mixture of 1—1/2% of hexamethylene tetramine and 3% of dicyclohexylamine has been found to retain its flexibility after fifty-six days' exposure through glass to Los Angeles, California summer sunshine. When not exposed to the direct rays of the sun it remains flexible for a much longer time and is entirely suitable for packaging cigarettes, cigars, edibles of all kinds such as bread, candies, etc. in boxes and individually and all sorts of articles for display purposes.

It appears that the change which occurs in the rubber hydrochlorides when exposed to the sunlight is some form of oxidation. Whatever the cause or nature of the change, the fact is that these photochemical inhibitors retard or inhibit some action induced or hastened by the sunlight which causes flexible rubber hydrochlorides which are not protected by an inhibitor to become brittle. The inhibitors appear to prevent a similar change from taking place in the more saturated tougher hydrochlorides when exposed to sunlight. In general, the inhibitors prevent a rapid decrease in the tear-resistance and tensile and impact strengths of flexible rubber hydrochloride films on exposure to sunlight. Certain of them also increase the life and otherwise affect the properties of rubber hydrochloride films, etc. used or stored in places where they receive little or no sunlight.

Moisture tests show that rubber hydrochloride films according to the invention are moisture-proof to a high degree. The tests were carried out in a box equipped with a fan to circulate the air over containers of 95% sulfuric acid in the box and maintain uniform humidity. The temperature was maintained at 39.5 deg. C. by thermostatic control. Each example of the film tested was laid on a glass plate. A 35 mm. diameter crucible containing 10cc. of water at 60 deg. C. was placed on the film. A crystallizing dish 70 mm. in diameter and 50 mm. deep was dipped about 5 mm. deep in melted beeswax and inverted over the crucible on the film. When the beeswax had hardened the dish was turned over, emptying the water from the crucible into the dish. The edges of the film were trimmed to about 8 mm. from the edge of the dish, slit at about 5 mm. intervals, and the edges fastened to the outer wall of the dish with beeswax.

The unit so prepared was weighed, placed in the box for a definite period, and then removed and re-weighed to determine the loss of water.

Many samples of rubber hydrochloride films about .001 inch thick containing different inhibitors have been tested in this box. In general the moisture transmission has been under 20 grams of water per square meter for 24 hours, and moisture transmission as low as 10 grams, and even 7 grams has been found in certain films. Tests run for two consecutive 24-hour periods show no increase in moisture transmission during the second twenty-four hour period. The moisture-proofness of a flexible rubber hydrochloride film is affected little, if any, by creasing. Seams of packages formed by heat-sealing the edges of the film together are substantially as moisture-proof as the film itself.

The addition of the photochemical inhibitors appears to have no radical effect on the flammability or moisture transmission of films. Plasticizers may affect these properties quite materially, without rendering the films unsuitable as wrapping materials. The judicious use of plasticizers may improve the film as a wrapping material for special purposes or for general use.

The partially saturated rubber hydrochloride may be prepared as follows: 20 pounds of plasticized pale crepe rubber are dissolved in about 313 pounds of benzene, giving a rubber cement of approximately 6% concentration. Commercial benzene containing up to 0.1% of water may be used. Dehydrated benzene gives a somewhat lighter colored film, although in either case a substantially colorless film may be obtained. The cement is preferably cooled to about 10 deg. C. and hydrogen chloride gas introduced, the cement being meanwhile agitated. The hydrogen chloride may be introduced at a higher temperature if desired, for example, at room temperature, but it is preferable to carry out the reaction at about 10 deg. C., as the gas is more readily dissolved in the cement at this temperature. After passing the gas into the solution for about six hours, the increase in weight of the composition due to the introduction of hydrogen chloride should be approximately 11.6 pounds which corresponds to a slight excess of available hydrogen chloride over that theoretically required by the empirical formula $(C_5H_9Cl)_x$, and when this increase in weight has been effected the introduction of hydrogen chloride gas into the cement is advantageously discontinued. If more hydrogen chloride is introduced the time required for the reaction of the hydrogen chloride and rubber is shortened but in most commercial operations this will not be advantageous.

The hydrogen chloride dissolved in the cement is allowed to react with the rubber at room temperature until a washed and dried sample indicates that the desired hydrochlorination has been effected. Generally about 20 hours will be required to produce 80 to 90% saturation of the rubber. Introducing an excess of hydrogen chloride ranging upward to about 50% over that theoretically necessary to obtain the desired partial saturation of the rubber is helpful in achieving the desired change in the rubber in a somewhat shorter time, but in general a prolonged period of standing will be found to be necessary. The test for completion of the desired saturation may be an analysis for chlorine or an empirical test such as a determination of the viscosity of the rubber hydrochloride or a determination of the stress strain properties of a film prepared from the reaction product or a visual and manual examination of a film prepared from the mass to determine whether or not it is tacky or brittle on drying.

After the desired partial saturation has been achieved, the excess hydrogen chloride may be neutralized with ammonia or other alkali and a photochemical inhibitor added directly to the neutralized solution and the wrapping material prepared from this. A somewhat clearer film which has longer life is obtained if water-soluble materials are first removed from the rubber hydrochloride.

Other methods may be employed for removing the excess hydrogen chloride, one being to bubble nitrogen through the reacted cement, the nitrogen taking the free hydrogen chloride with it. Another is to employ selective solvents. In some cases, it may be found desirable to remove the hydrogen chloride by precipitating it out as a salt. Still other methods of purification may be utilized if desired.

According to a preferred method of preparing a film, the reacted cement, after standing a sufficient time to effect the desired hydrochlorination of the rubber is subjected to steam distillation to remove the solvent and excess hydrogen chloride. The resulting mass of the partially saturated rubber hydrochloride is then broken up on a rubber washer, washed thoroughly with water to remove any remaining hydrogen chloride and water-soluble material and dried in a vacuum drier at approximately 160 deg. F. The dried product is then dissolved in chloroform, about 20 parts of solvent to one part of the

rubber hydrochloride being satisfactory. Other solvents, for example, benzene, may be employed if the materials are heated. Solvents such as dichlorethylene may be used without heating. To the resulting solution a suitable photochemical inhibitor is added. If the inhibitor is a mixture of compounds, the individual compounds may be added separately or together. The inhibitor may be added at an earlier stage of the process if it is not affected by the subsequent steps in the production of the film. It should be a high boiling compound and unless the film is to be colored as by the addition of dyestuffs, it should be colorless and a compound or mixture which, if it decomposes during the life of the film will not discolor the film. Aliphatic compounds and saturated ring compounds are preferred as inhibitors and secondary and tertiary aliphatic, alicyclic and heterocyclic amines, have been found very effective. Rubber hydrochloride films in which the following compounds have been incorporated in the proportions indicated, which are per cent by weight of the rubber hydrochloride, have been found to remain flexible much longer than films of the same rubber hydrochloride to which nothing has been added:

.5% Hexamethylene tetramine
1.0% ,,
1.5% ,,
3% Dicyclohexylamine
10% ,,
12% ,,
2% Ditetrahydrofurfurylamine
3% ,,
4% ,,
2% Methyleneaminoacetonitrile
3% ,,
2% Cyclohexylamine-formaldehyde
4% ,, ,,
6% ,, ,,
8% ,, ,,
10% ,, ,,
12% ,, ,,
2% Cyclohexylamino-acetonitrile
4% ,, ,,
5% ,, ,,
3% Tetramethyl thiuram disulfide
5% Diphenylguanidine
3% Dihydroxy diphenyl propane

The following mixtures of compounds have given satisfactory results:

1½% Hexamethylene tetramine
3% Methyleneamino-acctonitrile
.5% Hexamethylene tetramine
3% Ditetrahydrofurfurylamine
1% Hexamethylene tetramine
1% Ditetrahydrofurfurylamine
1% Hexamethylene tetramine
2% Ditetrahydrofurfurylamine
1% Hexamethylene tetramine
3% Ditetrahydrofurfurylamine
1½% Hexamethylene tetramine
1% Ditetrahydrofurfurylamine
1½% Hexamethylene tetramine
1% Ditetrahydrofurfurylamine
1½% Hexamethylene tetramine
2% Ditetrahydrofurfurylamine
1½% Hexamethylene tetramine
3% Ditetrahydrofurfurylamine
1½% Hexamethylene tetramine
1% Hexamethylene tetramine
3% Dicyclohexylamine
1½% Hexamethylene tetramine
1% Dicyclohexylamine
1½% Hexamethylene tetramine
2% Dicyclohexylamine
1½% Hexamethylene tetramine
3% Dicyclohexylamine
1½% Hexamethylene tetramine
1% Dicyclohexylamine
1% Ditetrahydrofurfurylamine
1½% Hexamethylene tetramine
1% Dicyclohexylamine
2% Ditetrahydrofurfurylamine
1½% Hexamethylene tetramine
2% Dicyclohexylamine
1% Ditetrahydrofurfurylamine
1½% Hexamethylene tetramine
1½% Dicyclohexylamine
1½% Ditetrahydrofurfurylamine

Cyclohexylamine formaldehyde may be prepared by crystallization from a solution of equimolar quantities of formaldehyde and cyclohexylamine in a solvent.

Films made by the addition of up to 5% of an inhibitor are considered to be composed almost entirely of a rubber hydrochloride, i.e. they contain at least 95% of the rubber derivative. Plasticizers, etc. may be added, if desired, in any suitable amount.

From the solution of the partially saturated rubber hydrochloride and photochemical inhibitor a film is prepared by any suitable means. Satisfactory results have been obtained by spreading the solution on a nitrocellulose-coated belt and then evaporating the solvent by circulating warm air over the surface, stripping the resulting film from the belt after substantially all of the solvent has been evaporated and subsequently evaporating the balance of the solvent. A belt with a coating of approximately equal parts of cellulose acetate and 2 : 1 ethylene glycolphthalic anhydride condensate may likewise be used. The solution may be sprayed or extruded onto any suitable smooth surface, such as glass, etc. in producing the film, if desired. The thickness of the film may be varied.

The film so produced is similar in appearance to the transparent cellulosic films sold under the registered Trade Mark "Cellophane". It is, however, much more resistant to moisture than are these films and for most purposes is therefore a more advantageous wrapping

material. The moisture-proof quality of many films in commercial use is reduced or destroyed when the films are wrinkled or creased, as for example, when the films are folded in wrapping an article. The film of the present invention is very flexible and is substantially as resistant to moisture after creasing and wrinkling as before.

It is to be understood that many variations may be made in the steps of the process of preparing the film and different materials may be employed. For instance, although pale crepe rubber will generally be found to be particularly suitable to the practice of the invention, other types of rubber may be employed. Benzene, chloroform, carbon tetrachloride, carbon bisulfide or other suitable solvents may be used in forming the rubber cement. Chloroform, dichlor ethylene and other solvents may be used in dissolving the rubber hydrochloride for the preparation of the film. The concentration of the rubber solution used in forming the film may be varied within reasonable limits. Although for wrapping purposes, a film .001 inch thick will be most suitable, films or sheets .0005 to .002 inches or thinner or thicker may be made.

An alternative method of producing the film comprises treating a seven per cent solution of pale crepe rubber in benzene at room temperature with dry hydrogen chloride gas which may advantageously be saturated with benzene, and continuing the treatment until the increase in weight is approximately five per cent. The introduction of hydrogen chloride is then discontinued and the cement is aged for a period of about twenty hours. The reacted cement is then poured into alcohol, allowed to harden, washed with water until all the hydrogen chloride is removed, and dried in a vacuum drier or other suitable apparatus. It is then dissolved in chloroform to form a seven per cent cement, to which two per cent of methylene amino aceto nitrile, based on the rubber hydrochloride content, are added. The film is formed from this cement in any desired manner.

Although in preparing the film from rubber hydrochloride the saturation of the rubber with hydrogen chloride may vary between 85 and 90%, a film which is approximately 88% saturated has been found to be unusually satisfactory and constitutes a preferred form of the invention. Rubber which has been incompletely saturated with other hydrohalides such as hydrogen bromide, also may be made into flexible films which may be stabilized as herein described to form wrapping materials. More saturated compounds may be mixed with paraffin, etc., to give a film of the required flexibility, etc. Materials such as paraffin, etc., may be added to a rubber hydrochloride of 29 to 30.5% chlorine content to form a film having increased resistance to water or to otherwise modify its properties. Waxes, resins, etc., may be added to other halogenated or hydrohalogenated rubber compounds in preparing the wrapping material of this invention.

A solution of the partially saturated rubber hydrochloride a photochemical inhibitor makes a very desirable composition for coating paper and other fibrous materials to make them moisture-resistant and oil-proof. A suitable inhibitor may be added directly to the reacted cement or neutralized cement to form such a coating composition. A clearer coating is formed by first steam-distilling the reacted cement to remove the solvent and excess hydrogen chloride, washing the resulting mass with water on a rubber mill, and then dissolving the washed product and flexibility-preserver in chloroform or other solvent.

Paper, textile fabrics, etc. may be dipped into the coating composition or the coating composition may be applied to the fabric on a spreader or other suitable device. Fabricated paper articles, such as containers, etc. may be sprayed with the coating composition or dipped into a batch of the coating composition. Paper coated with a stabilized, partially saturated rubber hydrochloride may be used in the manufacture of oil-proof containers, etc. Paper of suitable thickness coated on one or both sides, cut to the proper size, makes a very satisfactory milk-bottle cap.

Films of the stabilized rubber hydrochloride or a cement of the stabilized rubber hydrochloride may be employed for joining laminations of glass as in shatterproof windshields and the like.

It has been found that the photochemical inhibitors mentioned herein have a stabilizing effect on rubber derivatives of varying hydrochloride content. The invention is therefore not limited to the use of such compounds with flexible rubber hydrohalides, but includes the use of these compounds as stabilizers in saturated tougher compounds whether used alone or with plasticizers, etc.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed we declare that what we claim is :—

1. A composition of matter which comprises a halogen containing rubber derivative which contains one or more of the following compounds :

material. The moisture-proof quality of many films in commercial use is reduced or destroyed when the films are wrinkled or creased, as for example, when the films are folded in wrapping an article. The film of the present invention is very flexible and is substantially as resistant to moisture after creasing and wrinkling as before.

It is to be understood that many variations may be made in the steps of the process of preparing the film and different materials may be employed. For instance, although pale crepe rubber will generally be found to be particularly suitable to the practice of the invention, other types of rubber may be employed. Benzene, chloroform, carbon tetrachloride, carbon bisulfide or other suitable solvents may be used in forming the rubber cement. Chloroform, dichlor ethylene and other solvents may be used in dissolving the rubber hydrochloride for the preparation of the film. The concentration of the rubber solution used in forming the film may be varied within reasonable limits. Although for wrapping purposes, a film .001 inch thick will be most suitable, films or sheets .0005 to .002 inches or thinner or thicker may be made.

An alternative method of producing the film comprises treating a seven per cent solution of pale crepe rubber in benzene at room temperature with dry hydrogen chloride gas which may advantageously be saturated with benzene, and continuing the treatment until the increase in weight is approximately five per cent. The introduction of hydrogen chloride is then discontinued and the cement is aged for a period of about twenty hours. The reacted cement is then poured into alcohol, allowed to harden, washed with water until all the hydrogen chloride is removed, and dried in a vacuum drier or other suitable apparatus. It is then dissolved in chloroform to form a seven per cent cement, to which two per cent of methylene amino aceto nitrile, based on the rubber hydrochloride content, are added. The film is formed from this cement in any desired manner.

Although in preparing the film from rubber hydrochloride the saturation of the rubber with hydrogen chloride may vary between 85 and 90%, a film which is approximately 88% saturated has been found to be unusually satisfactory and constitutes a preferred form of the invention. Rubber which has been incompletely saturated with other hydrohalides such as hydrogen bromide, also may be made into flexible films which may be stabilized as herein described to form wrapping materials. More saturated compounds may be mixed with paraffin, etc., to give a film of the required flexibility, etc. Materials such as paraffin, etc., may be added to a rubber hydrochloride of 29 to 30.5% chlorine content to form a film having increased resistance to water or to otherwise modify its properties. Waxes, resins, etc., may be added to other halogenated or hydrohalogenated rubber compounds in preparing the wrapping material of this invention.

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Films of the stabilized rubber hydrochloride or a cement of the stabilized rubber hydrochloride may be employed for joining laminations of glass as in shatterproof windshields and the like.

It has been found that the photochemical inhibitors mentioned herein have a stabilizing effect on rubber derivatives of varying hydrochloride content. The invention is therefore not limited to the use of such compounds with flexible rubber hydrohalides, but includes the use of these compounds as stabilizers in saturated tougher compounds whether used alone or with plasticizers, etc.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed we declare that what we claim is :—

1. A composition of matter which comprises a halogen containing rubber derivative which contains one or more of the following compounds :

* C L M S *

Hexamethylene tetramine
Dicyclohexylamine
Ditetrahydrofurfurylamine
Methyleneaminoacetonitrile
Cyclohexylamine-formaldehyde
Cyclohexylaminoacetonitrile
Tetramethyl thiuram disulphide
Diphenylguanidine
Dihydroxy diphenyl propane.

2. A composition of matter as claimed in claim 1, which comprises a partially saturated rubber hydrochloride.

3. A composition of matter as claimed in claim 2, in which the rubber hydrochloride contains 29% or more of chlorine.

4. A composition of matter as claimed in any of the claims 1—3, in which the halogen containing rubber derivative is prepared by hydrochlorinating rubber in the presence of a solvent, and then removing the solvent and impurities.

5. A compositions of matter as claimed in claim 4, wherein impurities are removed by washing.

6. The improved compositions of matter substantially as described.

7. Articles consisting of or comprising the composition of matter claimed in any one of more of the preceding claims.

8. Articles according to claim 7, which comprise material, e.g. paper, fabric impregnated or coated with the composition of matter.

9. Material in sheet form which consists of or comprises the composition of matter claimed in any one or more of claims 1 to 6.

10. Wrapping material which consists of or comprises the composition of matter claimed in any one or more of claims 1 to 6.

11. Wrapping material as claimed in claim 10, in which the composition of matter forms a coating for a sheet-formed base.

12. Wrapping material as claimed in claim 10, or 11, which is transparent.

13. Safety glass comprising laminations of glass joined together with the aid of the composition of matter claimed in any one or more of claims 1 to 6.

14. The improved wrapping material substantially as hereinbefore described.

Dated this 22nd day of January, 1934.

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Abingdon: Printed for His Majesty's Stationery Office, by Burgess & Son.
[Wt. 8031B —50/7/1936.

