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## ELASTO-PLASTIC WINDOW GLAZING COMPOUND

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The present invention pertains to improvements in glazed window sash and glazing compounds adapted for such use. The type of glazing compounds especially contemplated are those of elasto-plastic nature, for example comprising rubber and/or various rubbery materials including such synthetic materials as "Vistanex," polyvinyl butyral resins, "Thiokol" (a diolefine polysulfide type compound), "Neoprene," butyl rubber, and the like.

Heretofore the common material employed in glazing window sash has been the ordinary linseed oil and ground calcium carbonate putty, or in some cases white lead. Ordinary putty has a number of well recognized disadvantages, among which are its tendency to shrink and pull away from the sash and/or glass, brittleness and tendency to crack and fall off, thus leaving the seam unprotected, etc.

It is an object of this invention to provide a window sash of an improved nature over sash glazed with the types of putty or glazing compounds heretofore employed. A further object of this invention is to produce glazed sash in which the glazing material is of elasto-plastic character, so that it will yield internally with such distortion or warping of various parts of the sash as may occur during handling or use, and yet will still firmly adhere to the glass and to the wood, metal or other type of material out of which the sash frame is made. A further object is to provide a glazing compound which can be applied with a glazing gun, as distinguished from a compound adapted only to be applied with a putty knife or glazing knife. A further object is to provide a glazing compound which will maintain an approximately uniform viscosity during storage, can readily be forced through a relatively small orifice or opening of a glazing gun by air or hydraulic pressure during glazing at ordinary room temperatures, has the property of adhering and remaining in place on vertical seams or surfaces while drying, and which has other advantages. These and other objects and advantages will appear from the specification taken as a whole.

In the manufacture of glazing cements, especially those adapted for use in glazing window sash, where the surface of the glazing compound is commonly exposed to the weather during use, many different features have to be considered. Not necessarily listed in order of importance, nor even completely, they may be stated as comprising reasonable cost; ease in handling; adaptability to be applied to desired surfaces or seams by air pressure, such as may be applied with a glazing gun, as above suggested; ability to remain in place, and to dry at a speed which will permit of storage or shipment of the glazed sash unhampered by too delicate precaution; good adhesion to glass, wood and metal, or the like; freedom from sagging and sliding during application to horizontal or vertical seams and/or putty rabbetts; drying without substantial checking, cracking or pulling away from the glass, wood, or like surfaces; imperviousness to water seepage, both in the body of the glazing composition and at the points of contact with glass, wood and metal; and, ability to withstand higher than normal sun temperatures and quickly alternating extremes of heat and cold.

Many of these requirements are not met by the putties in common use, due to weakness such as absorption by the wood of the oils used as plasticizer, thus resulting in weakness of the bond at the juncture with subsequent detachment of the putty.

By the use of a proper combination of such materials as rubber, resins such as rosin or the like, filler material, fibre and solvent, I have found that it is possible to obtain a more intimate bond between the glass and wood, or glass and metal, and the like, than was possible with prior art putties known to me. The evaporation of the solvent should be fast enough to permit a desired rate of drying and firming, but slow enough to inhibit or prevent bubbling and blistering.

Illustrative of various materials which may be used in forming my elasto-plastic glazing compositions, are reclaimed rubber, such as whole tire reclaim and inner-tube reclaims, raw rubber, melted scrap inner-tubing, certain synthetic rubbers, certain rubbery synthetic resins, and the like. All such materials are characterized by their elasto-plastic nature and may be referred to as elasto-plastomers.

Melted scrap inner-tubing is, in general, less desirable than such forms of rubber as reclaimed rubber. However, melted inner-tubing does have the virtue of requiring less solvent and has some advantage where a very high solids content is desired but, on the other hand, the glazing compound made therewith, unless bolstered with other forms of rubber, does not firm up to the same degree as, for example, compounds made with reclaimed rubber, and various synthetic rubbery compounds.

Where reclaimed rubber is employed, it is desired to have the same substantially free from fibre bundles, especially where the glazing compound is to be applied with a glazing gun under air-pressure (according to the preferred mode of applying by glazing compound) in order that the gun will not be obstructed or clogged or application otherwise impaired. However, absence of such fibre bundles or "unbroken down" fibre is desirable even where the same is to be applied with a knife.

In respect to the resin content of the composition, as for example where reclaimed rubber is employed, I have employed wood-rosin such as "Solros" (manufactured by General Naval Stores) and also have used other resins including those of the coumarone-indene type, e. g. cumar and like resins, which are derivable from coal tar naphtha. I have also employed "Nevillite" resin (a resin which may be derived from coumarone-indene type resins by hydrogenation, e. g. with Raney Nickel catalyst, and made and sold by the Neville Company, Pittsburgh) along with forms of rubber including reclaimed rubber. The same is advantageous, but, at the present time, is relatively costly for use in window glazing compounds. (For descriptions of hydrogenation techniques, in this connection, see U. S. patent to Carmody, No. 2,152,533, issued March 28, 1939, and Industrial and Engineering Chemistry, vol. 32, pages 684–92, May, 1940.)

I have also employed modified resin such as zinc treated and lime treated rosins, such as so-called "Zitro" and "Fosfo" (sold by General Naval Stores) but, in general, such metallic rosinates have not been as advantageous as rosin itself, and certain other resins, above discussed.

The selection of a satisfactory filler from such materials as Dixie clay (a kaolin clay of a relatively large particle size compared with bentonite), "Asbestine" (a variety of talc or hydrated magnesium silicate of needle-like crystalline structure), slate flour, and whiting (calcium carbonate), and other like materials presented difficulties, and further difficulties were encountered in determining the quantities desirable to give satisfactory body to the composition without producing a punky, non-cohesive mass with poor application qualities (especially in a glazing gun) and an adhesion to glass, wood and metal below that desired under normal drying conditions, and especially under the more rapid or drastic condition of drying sometimes encountered. Whiting was found, in general, all things considered, to be the most satisfactory of a large number of filler materials tested in various formulations. In many formulations, slate flour showed a tendency toward "gumbo" formation and "Asbestine" showed a "shortness" attended by a somewhat slimy condition. Dixie clay showed certain advantages as a filler but comparatively large proportions thereof were ordinarily needed to inhibit or obviate blistering under extreme conditions. Such quantities of Dixie clay, or the like, however, ordinarily necessitated a proportionately larger quantity of solvent and this, in turn, created a problem in avoiding a "stringy" product. The use of asbestos fibre materially helped this phase and it was found that prolonged agitation of the mass in an internal mixer (e. g. of the Werner-Pfleiderer type or Baker-Perkins type) subsequent to the addition of the asbestos, helped to reduce or inhibit sagging or flowing of such a cement after application to window sash. However, even with the use of asbestos fibre or equivalent, I still prefer a filler material having a substantially larger particle size than Dixie clay, but of a smaller particle size than slate flour, whiting being a preferred filler in my composition.

It is desirable in the commercial production of my glazing compositions, where asbestos fibre is employed, to mill the asbestos and rubber together on a rubber mill, before introducing the mass into an internal mixer. Otherwise, it is difficult to control other qualities of the glazing composition as desired without ending up with a compound which tends to be somewhat dry and to have a somewhat reduced adhesion to wood, glass and metal, and also has some tendency toward permitting cracking of the glazing compound, especially at the corners of the sash. The Dixie clay also appeared to be responsible for a "livering" condition, encountered in various formulations, which was obviated by the use of whiting in place of Dixie clay. The use of whiting, or other suitable filler, of comparable particle size, along with suitable quantities of other ingredients, particularly those hereinabove discussed, finally led to a product of desired "short" and "buttery" consistency, but which flows freely from a glazing gun tip (e. g. of ⅛ inch in diameter), and flows without that "stringy" quality which makes satisfactory application practically impossible, particularly at the corners of the sash. The corners of the sash are one location, above all others, where a good bond should be attained.

The use of a relatively high boiling point solvent is favored in that it makes for easy handling of the glazing composition, with the danger of skimming over (due to rapid evaporation of the solvent) eliminated or substantially so. Too rapid evaporation of the solvent was also found to result in cracks or pinholes in the composition on the glazed sash. Suitable solvents will be illustrated hereinbelow, rate of evaporation and cost being important considerations.

Various compositions employing waxes were experimented with, in a search for extreme waterproofness. However, the waxes, though lending water-resisting qualities to the glazing compositions, slowed up drying and firming of the glazing composition to an extent that was undesirable for most production uses in the glazing of sash. It was therefore desirable to develop a composition of good waterproof qualities in the absence or substantial absence of such an inhibitor of drying and firming.

The following is illustrative of one of the early compositions developed to provide an elasto-plastic glazing composition.

*Formula I*

| | |
|---|---|
| Red-inner tube reclaim ............ lbs. | 400 |
| Limed FF wood rosin (4 lbs. lime for 100 lbs. rosin) ............ lbs. | 600 |
| Finely divided kaolin ............ lbs. | 800 |
| Short fibre asbestos ............ lbs. | 260 |
| A petroleum hydrocarbon material having a distillation range of 90° to 120° C., 194° to 248° F ............ gals. | 160 |

In this connection reference is made to Example 6 appearing on page 8 of the co-pending application of Merrill et al., Serial No. 262,176, filed March 16, 1939. The present application, insofar as it possesses related or common disclosure, is a further development or continuation-in-part of the aforesaid application.

Though the composition shown in the above formula has decided advantages over linseed oil putties and the like, I have found that by the use of different formulations I am able to produce glazing compositions which are appreciably superior, both in respect to their use during glazing of sash and in respect to the resulting glazed sash. An improved composition which I have developed is as follows:

*Formula II*

| | Pounds |
|---|---|
| Red inner-tube reclaim (neutral or of low alkalinity) | 400 |
| Wood rosin ("Solros") | 600 |
| Whiting (powdered calcium carbonate) | 400 |
| Asbestos fibre | 200 |
| Hydrocarbon solvent (boiling range 305°–365° F.) | 596 |

The composition of Formula II was superior to that of Formula I, among other things, in resisting, in the dried state, cracking at the corners of the window sash and tendency to pull away from the glass, wood or metal during drying and aging. It is also superior in that its viscosity aging is much better; that is, it maintains an approximately constant viscosity throughout a prolonged storage, in containers, prior to use. These improvements are of highly important consequence, as will be readily appreciated from what has been said hereinabove with regard to the desirable and necessary qualities in successful glazing cement compositions. From what has been said hereinabove, it will be observed that (1) the use of rosin, instead of limed rosin; (2) the use of whiting in place of kaolin; and (3) the use of a solvent which evaporates less rapidly, are features of composition which contribute to the improved qualities of the composition of Formula II.

Because of the various qualities desired and needed in a glazing composition of this kind, as will be evident from the discussion given hereinabove, it will be observed that the difference in effect of whiting over Dixie clay, for example, influences various other qualities which must be otherwise compensated for in the formulation of glazing cement. Whereas 800 lbs. of kaolin (e. g. Dixie clay) were employed in Formula I with 400 lbs. of rubber, an appreciably lower amount of whiting was employed in Formula II in relation to the rubber. This is simply illustrative.

A further composition which I have developed, having advantages over both of the compositions of Formulae I and II, is as follows:

*Formula III*

| | Pounds |
|---|---|
| Red inner-tube reclaim (neutral or of low alkalinity) | 800 |
| Wood rosin ("Solros") | 400 |
| Cumar (M. P. 125°–135° C.) | 800 |
| Whiting (powdered calcium carbonate) | 800 |
| 7 R. asbestos fibre (short fibre) | 400 |
| Hydrocarbon solvent (305°–365° F. boiling range, spec. grav. 0.757) | 1,514 |

The composition of Formula III has the following further advantages over that of Formula II: (1) It is more resistant to the development of sun-check lines; (2) It is somewhat faster setting, which is a production advantage; (3) It is tougher in the dried state (after evaporation of the solvent) and the resulting sash is more durable as a consequence; and (4) It is somewhat more buttery, more free of "stringy" tendency, which is an advantage in application to the putty rabbett of window sash, doors and the like. It is to be observed that regular putties are not considered satisfactory on doors, one all-sufficient reason being their lack of shock resistance.

While the compositions above set forth may be formulated in various ways, one suitable and advantageous method of formulation (in connection with which the composition of Formula II may be especially considered) is as follows: The rubber and asbestos fibre are milled together on a rubber mill, preferably only long enough (about 10 or 20 minutes) uniformly to disperse the fibre in the rubber. The resulting blended rubber and asbestos fibre may then be taken off the rubber mill and introduced into an internal mixer e. g. of the Baker-Perkins type. The whiting and rosin are then gradually added to the rubber in increments with continued mixing, the whiting and the rosin being added alternately. Each of these ingredients may be added as fast as permissible without breaking up the coherency of the mass. For example, about one-half the whiting, or slightly less, may be added and, after several minutes of mixing, about one-half of the rosin or other resins, or slightly more, may be added. Then, after several more minutes, the remainder of the whiting may be added and about five minutes later the remainder of the rosin or other resin may be added. An important thing throughout the mixing is, on the one hand, to avoid getting too much molten resin present at one time in order to prevent the batch breaking up into slippery lumps which do not readily reunite into the desired doughy condition and sometimes create a permanently lumpy condition in the finished cement and, on the other hand, to avoid getting so much whiting or comparable filler into the batch at one time that it becomes crumbly and lacing in coherency, thereby to maintain a proper balance to promote good mixing. Ordinarily no heat need be added or withdrawn during the foregoing mixing operation. The rubber and asbestos blend is warm when it comes from the rubber mill and the mechanical mixing ordinarily generates about the right amount of heat to promote good mixing. However the temperature can be controlled, if desired, by the addition of either cold water or steam to the jacket of the internal mixer.

After the solids of the composition are uniformly mixed together to provide a substantially uniform blend, the solvent may then be gradually added. The solvent may be added, for example, in three or four separate increments, the introduction of each being spaced by enough time (10 or 15 minutes for example) so as to get uniform mixing without breaking up the coherency of the mix. Ordinarily enough solvent is added to bring the viscosity of the desired glazing cement to, for example, approximately 3 minutes at 75° F., where the viscosity is measured by the time required for one-half pint of glazing composition to pass through a pipette type viscosimeter having a pipe 8 inches long and of one-fourth inch diameter, under a pressure of 60 lbs./sq. in. gauge.

The composition of Formula III, as well as that of Formula I, may be formulated according to generally the same method. In the composition of Formula III, the cumar and "Solros" resins may be mixed together and added in increments, or increments of each material may be added separately, as desired.

It will be understood that the above formulae are given by way of illustration and not by way of limitation. It will be evident that the nature of the solvent may be varied within limitations so long as the desired drying characteristics of the glazing compositions are not substantially hindered. Likewise, as above indicated, in place of various types of ordinary rubber, various rubbery vinyl resins, such as various butyral resins, synthetic rubbery compositions such as "Vistanex," etc., may be employed. Likewise wood rosin may be replaced wholly or in part by such materials as ester gum, "Nevillite" resin, and various other natural and syntheic resins. As illustrated in Formula III, such resins as cumar may be employed to advantage along with such a resinous material as wood rosin. Cumar and various other resins having the property of not retaining solvent as tenaciously as wood rosin, and thus may be used to advantage. However such resinous materials, to be suitable, must also be good solvents for, or must be highly compatible with, rubber. In place of whiting other fillers of comparable particle size may be used. Clays such as Dixie clay are of much smaller particle size than whiting, and material such as slate flour are of much larger particle size. Other fillers of particle sizes comparable to that of whiting are illustrated, for example, by such materials as blanc-fixe, barytes, or other common rubber and paint fillers of sizes comparable to whiting. In view of the cheapness and ready availability of whiting, it is commonly preferred. In place of asbestos fibre, various natural vegetable, mineral and synthetic fibres may be used, cotton linters and "nylon" being examples.

Some attempts have previously been made to make rubber-containing glazing compositions for use as so-called "knife coating compositions," but not at all adapted for use in a glazing gun. While my glazing cements may be applied with a knife, a feature of prime importance is that they are adapted for application with a glazing gun; that is, they are of viscosity such that they can be forced through a small opening (e. g. one-eighth inch diameter) of a glazing gun under pressure and yet have sufficient body, and other properties, so that, when applied to the putty rabbett of window sash and doors, they will remain in place without sagging.

Sash and doors glazed with the preferred compositions hereinabove described may be shipped within about 2 days after application of the glazing composition. Whereas sash glazed with ordinary linseed oil putties have a tendency to wrinkle as a result of slight distortion of the sash during handling in shipping, sash glazed according to the present invention are free of such tendencies, particularly in view of the elasto-plastic nature of my glazing composition.

All embodiments of this invention are comprehended within the scope of the following claims.

What I claim is:

1. An elasto-plastic sash glazing composition having good adhesion to wood, glass and metal and of such viscosity that it can readily be applied at ordinary room temperatures to the putty rabbett of a sash by forcing the same under pressure through a confined passage of a glazing gun, and further of such body that it will not sag appreciably after application and during drying in a vertical putty rabbett, said glazing composition comprising reclaimed rubber, wood rosin, cumar, powdered whiting and asbestos fiber, dispersed in a hydrocarbon solvent having a boiling range of the order of 305°–365° F., said reclaimed rubber being present in greater proportion than said rosin but in lesser proportion than the combined resinous material, said powdered whiting being present in substantial but lesser proportion than the combined rubber and resin content, and the asbestos fibre being present in substantial but lesser amount than said whiting.

2. An elasto-plastic sash glazing composition having good adhesion to wood, glass and metal and comprising an intimate blend of ingredients of the kind and substantially in the proportion as follows:

| | Pounds |
|---|---|
| Reclaimed rubber | 800 |
| Wood rosin | 400 |
| Cumar resin (M. P. 125°–135° C.) | 800 |
| Powdered whiting | 800 |
| Short fibre asbestos | 400 |
| Hydrocarbon solvent (305°–365° F. boiling range and specific gravity of the order of 0.757) | 1,514 |

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