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# PATENT SPECIFICATION

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## (54) FLAME-RESISTANT SEALING COMPOSITIONS

(71) We, HERBERTS GMBH., a body corporate organised according to the laws of the Federal Republic of Germany, of 25 Christbusch, D-5600, Wuppertal 2, Federal Republic of Germany, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

The present invention relates to sealants.

Flame resistant paints forming an insulating layer are known. They are applied as normal paints on to construction pieces of steel, concrete or wood, and in the event of fire, they form a porous, inflated, heat insulating layer which retards the heating of the substrate such that the stability of steel or concrete parts is lost much later than that of unprotected pieces and the combustion of wood is delayed. However, such paints are suitable for the protection of plane surfaces only.

Various systems which are intended to provide fire-resistant sealing of joints, for example expansion joints in ceilings, or for securing openings or cavities, have been proposed. These include, for example, glass fibre laminates, combinations of glass fibres or foamed plastics granules bound by butyl rubber strips (British Patent Specification No. 1,080,170), sealing compositions made from reaction products of sulphur with diphenyldithiophosphate and styrene (French Patent Specification No. 1,502,687), and vulcanized silicon elastomers (Japanese Patent Specification No. 094,385).

To some extent, however, these compositions are only suitable for sealing gaps, and not for securing large cavities. Although they have the common property of being flame-retarding or nonflammable, they do not form any insulating layer on combustion and they become brittle and lose their adhesion, so that a secure seal is no longer ensured.

We have now discovered a sealing composition which forms an insulating layer and which does not embrittle over a long period of time. In the event of fire, the sealed gaps and cavities remain fire-proof and smoke-tight.

The present invention provides a sealing composition which comprises one or more substances which have heat insulating properties, for example ammonium polyphosphate, melamine, pentaerythritol or starch or a mixture of any two or more of these compounds, and a binder which comprises:

(i) a vinyl chloride copolymer having a K value greater than 60, or a mixture of two or more such copolymers, and
(ii) a chlorine-containing polymer which is not a vinyl chloride copolymer or is a vinyl chloride copolymer having a K value in the range of from 50 to 60, or a mixture of two or more such polymers, and
(iii) at least one plasticizer which is capable, in the quantity employed, of dissolving and gelling the mixture of polymers (i) and (ii), at the temperature at which the ingredients are mixed, and
(iv) at least one plasticizer which does not dissolve the mixture of polymers (i) and (ii).

In addition, the binder may comprise any suitable stabilizer for the polymer mixture, or a mixture of stabilizers, for example an organic tin compound, or a mixture of such compounds.

A sealing composition of the invention may also comprise one or more inorganic fillers, for example calcium carbonate, barium sulphate, glass fibres or other mineral fibres or a mixture of two or more of these fillers. Such fillers affect the ease of processing and the stability of the composition.

Suitable vinyl chloride polymers (i) and/or (ii) are, for example, copolymers of vinyl chloride (VC) and maleic acid esters, especially dibutyl maleate, or vinyl acetate (VAC), which are commercially obtainable as raw materials for the lacquer and plastics industries. Another suitable chlorine-

containing polymer (ii) is, for example, polychlorobutadiene.

Suitable plasticizers (iii) which dissolve and gel the polymer mixture are, for example, aliphatic or aromatic esters of phthalic, adipic, benzoic, sebacic or phosphoric acid or a mixture of two or more such esters, or relatively non-volatile, chlorinated paraffins having a chlorine content of at least 50%. Phosphoric acid esters and halogenated hydrocarbons which act against the flammability of the composition are preferred.

Suitable plasticizers (iv) which do not dissolve the polymer and tend to exude and which help to maintain the composition in a durable plastic state are, for example, non-drying fatty acid esters, for example castor oil, chlorinated paraffins having a chlorine content of substantially 40% by weight trichloro-ethyl-phosphate, mineral oils, or dibenzyltoluene, or a mixture of two or more such compounds.

The sealing compositions of the invention need not contain any volatile substances. They have good adhesion to smooth substrates. They may be applied manually or by a blow gun; in the latter case, the application temperature should not exceed about 90°C.

The period of fire resistance of a sealing composition of the invention according to German Industrial Standard DIN 4102 is longer than 90 minutes. During a fire, the composition forms a heat insulating, mechanically stable protective layer consisting largely of carbon cells filled with gas. This layer is non-flammable. It protects the substrate material from heating and forms an efficient barrier against the penetration of smoke gases.

The following Examples illustrate the invention. All Figures given are parts by weight and percentages are by weight (except the K values, which are purely numerical).

## EXAMPLE 1

Ready-to-use sealing composition:

| | |
|---|---|
| ammonium polyphosphate | 15.7 |
| melamine | 14.0 |
| pentaerythritol | 10.6 |
| starch | 5.0 |
| calcium carbonate | 10.0 |
| barium sulphate | 9.7 |
| mineral fibre | 3.0 |
| binder | 32.0 |
| | 100.0 |

wherein the binder has the composition:

| | |
|---|---|
| diethylhexyl phthalate | 28.0 |
| tricresyl phosphate | 33.0 |
| chlorinated paraffin (approx. 50% chlorine) | 5.0 |
| epoxidised fatty acid | 3.0 |
| organic tin compounds | 0.5 |
| VC copolymer having a K value of 56—58, 80% VC and 20% various maleic acid esters | 12.0 |
| VC copolymer having a K value of approx. 65, 90% VC and 10% VAC | 5.0 |
| castor oil | 13.5 |
| | 100.0 |

## EXAMPLE 2

Ready-to-use sealing composition:

| | |
|---|---|
| ammonium polyphosphate | 14.6 |
| melamine | 13.2 |
| pentaerythritol | 10.0 |
| starch | 4.7 |
| calcium carbonate | 9.4 |
| barium sulphate | 9.3 |
| mineral fibre | 2.8 |
| binder | 36.0 |
| | 100.0 |

wherein the binder has the composition:

| | |
|---|---|
| diocytl sebacate | 30.0 |
| trichloroethyl phosphate | 30.0 |
| diethylhexyl phosphate | 5.0 |
| epoxidized fatty acids | 3.0 |
| organic tin compounds | 0.5 |
| VC copolymer having a K value of 55, 60% VC and 40% VAC | 13.0 |
| VC copolymer having a K value of 65, 90% VC and 10% VAC | 5.0 |
| castor oil | 13.5 |
| | 100.0 |

## EXAMPLE 3

Ready-to-use sealing composition:

| | |
|---|---|
| ammonium polyphosphate | 16.0 |
| melamine | 14.5 |
| pentaerythritol | 11.0 |
| starch | 5.5 |
| calcium carbonate | 11.0 |
| barium sulphate | 11.0 |
| mineral fibre | 3.0 |
| binder | 28.0 |

wherein the binder has the composition:

| | |
|---|---|
| trichloroethyl phosphate | 30.0 |
| benzylbutyl phthalate | 20.0 |
| dibutyl phthalate | 18.5 |
| epoxidized fatty acids | 3.0 |
| organic tin compounds | 0.5 |
| VC copolymer having a K value of 65, 85% VC | 10.0 |
| Polychlorobutadiene | 3.0 |
| castor oil | 15.0 |
| | 100.0 |

WHAT WE CLAIM IS:—

1. A sealing composition which comprises one or more substances which have heat insulating properties, and a binder, which binder comprises:
   (i) a vinyl chloride copolymer having K value greater than 60, or a mixture of two or more such copolymers, and
   (ii) a chlorine-containing polymer which is not a vinyl chloride copolymer or is a vinyl chloride copolymer having a K value in the range of from 50 to 60, or a mixture of two or more such polymers, and
   (iii) at least one plasticizer which is capable, in the quantity employed, of dissolving and gelling the mixture of polymers (i) and (ii), at the temperature at which the ingredients are mixed, and
   (iv) at least one plasticizer which does not dissolve the mixture of polymers (i) and (ii).

2. A sealing composition as claimed in claim 1, which contains as the substance or substances with heat insulating properties, ammonium polyphosphate, melamine, pentaerythritol, or starch, or a mixture of any two or more of these compounds.

3. A sealing composition as claimed in either claim 1 or claim 2, wherein component (i) comprises a vinyl chloride-maleic acid ester copolymer.

4. A sealing composition as claimed in claim 3, wherein component (i) comprises a vinyl chloride-dibutyl maleate copolymer.

5. A sealing composition as claimed in any one of claims 1 to 4, wherein component (i) comprises a vinyl chloride-vinyl acetate copolymer.

6. A sealing composition as claimed in claim 1, wherein component (ii) comprises a vinyl chloride-maleic acid ester copolymer.

7. A sealing composition as claimed in claim 1, wherein component (ii) comprises a vinyl chloride-dibutyl maleate copolymer.

8. A sealing composition as claimed in any one of claims 1 to 5, wherein component (ii) comprises a vinyl chloride-vinyl acetate copolymer.

9. A sealing composition as claimed in any one of claims 1 to 5, wherein component (ii) comprises polychlorobutadiene.

10. A sealing composition as claimed in any one of claims 1 to 9, wherein component (iii) comprises an ester of phthalic, adipic, benzoic, sebacic or phosphoric acid, or a mixture of two or more such esters.

11. A sealing composition as claimed in any one of claims 1 to 9, wherein component (iii) comprises a chlorinated paraffin having a chlorine content of at least 50%.

12. A sealing composition as claimed in any one of claims 1 to 11, wherein component (iv) comprises a non-drying fatty acid ester, a chlorinated paraffin having a chlorine content of substantially 40% by weight, trichloroethyl phosphate, a mineral oil or dibenzyltoluene, or a mixture of two or more such compounds.

13. A sealing composition as claimed in claim 12, wherein component (iv) comprises castor oil.

14. A sealing composition as claimed in any one of claims 1 to 13, wherein the binder also contains a stabilizer or mixture of stabilizers for the mixture of polymers (i) and (ii).

15. A sealing composition as claimed in claim 14, which contains as stabilizer an organic tin compound or a mixture of such compounds.

16. A sealing composition as claimed in any one of claims 1 to 15, which also contains an inorganic filler or mixture of inorganic fillers.

17. A sealing composition as claimed in claim 16, which contains as filler calcium carbonate, barium sulphate, or mineral fibres, or a mixture of two or more such fillers.

18. A sealing composition as claimed in claim 1, substantially as described in any one of Examples 1 to 3 herein.

19. A method for sealing a cavity, which comprises applying to the cavity a sealing composition as claimed in any one of claims 1 to 18.

20. A method for sealing a cavity as claimed in claim 19, wherein the sealing composition is applied using a blow-gun.

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