

REDISPERSIBLE DRY COMPOSITION OF POLYMER FINISHES OR OTHER PRODUCTS FOR THE CONSTRUCTION

Field of the Invention

5 The technical field of the invention relates to plasters, renderings, grouts, waterproofing coatings or mortars notably for the interior or exterior of buildings, such as finishes or mortars for the external thermal insulation as well as joints for the construction.

In particular, the invention concerns water-dispersible powder building material compositions useful for the preparation of wet formulations of plasters or mortars for the
10 construction and the civil engineering industry.

More particularly, the invention refers to cement-free and water-dispersible powder building material compositions comprising a homopolymer or a copolymer as binder.

The invention concerns also the methods of preparation of these dry compositions and wet formulations, the application of these latter onto the surface of buildings or civil
15 engineering works, the so obtained coatings as well as the so coated elements. The applications of these wet formulations can be also joints or other products for the construction as well as mass objects.

Background Art

20 Many liquid aqueous acrylic finishes are known. These are wet formulations containing an entirely organic binder made of an acrylic or styrene/acrylic or ethylene/vinylacetate polymer dispersion, a coalescent agent, a water retentive agent such as a cellulosic ether, a dispersant (e.g. a surfactant), a defoamer, a biocide, a carbonate sand and/or a silica sand, a carbonate filler, TiO_2 , a mineral or organic thickener, a pH regulator, exogenous water and
25 water from the polymer dispersion.

The acrylic or styrene/acrylic polymer dispersion of these acrylic finishes are obtained by emulsion polymerisation.

These known acrylic finishes are ready-to-use pastes which are applied on the exterior surfaces of buildings.

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One knows moreover redispersible powders of acrylic or styrene/acrylic polymer. These powders are produced by spray drying of a dispersion of this acrylic or styrene/acrylic polymer, including a water-soluble protective colloid such as polyvinyl alcohol and/or polyacrylates and/or polyvinyl pyrrolidone. See notably EP-723975-A and US5252704.

35 These powders can be used in mortar formulations containing a mineral binder (cement and/or lime).

One of the drawbacks of these cement-free pastes is that they badly withstand the frost and especially the cycles frost/thaw and more generally the thermal shocks.

Other drawbacks linked to the aqueous character of these known acrylic finishes, is also that:

- 5 (i) The water represents an additional cost in terms in of transport.
- (ii) The water imposes to load the pasty acrylic finish with high levels of biocide.

These features (i) and (ii) make these known acrylic finishes not really environment friendly.

10 **Summary of the invention**

The present invention pursues at least one of the following objects.

It is an object of the invention to develop redispersible dry compositions of polymer finishes or of other products for the construction which would not have the drawbacks of the known aqueous pasty acrylic finishes or construction products.

- 15 It is an object of the invention to develop redispersible dry compositions of polymer finishes which would not be prone to induce efflorescence on the surface of plasters, renderings, coatings made from this composition.

It is an object of the invention to develop redispersible dry compositions of polymer finishes which would not have a highly basic pH after being redispersed into water, to

- 20 manufacture wet formulations to be applied onto the surface of buildings or civil engineering works.

It is an object of the invention to develop redispersible dry compositions of polymer finishes which would not have an adverse effect on the processing properties (easiness and reproducibility of the mixing-workability-pumpability-non-tackiness-shotcreting-...)

- 25 the mechanical characteristics (water-resistance-hardness-durability-cracking strength-..) of the hardened plasters, renderings, coatings made from these compositions.

It is an object of the invention to develop redispersible dry compositions of polymer finishes which could be mixed with water to manufacture wet formulations to be applied onto the surface of buildings or civil engineering works, to form hardened plasters,

- 30 renderings or coatings, with protection and waterproofing properties.

It is an object of the invention to develop plasters, renderings, coatings with waterproofing and good-mechanical properties as well as protection, said plasters, renderings, coatings being obtained from dry compositions of polymer finishes easily redispersible in water.

- 35 It follows that the invention pertains to a redispersible dry composition of polymer finish or of other products for the construction comprising:

- a) a powdery organic binder including a polymer obtained by spray-drying or lyophilisation of a dispersion containing said polymer and a protective colloid;
- 5 b) a deprotection agent chosen among those which are effective without increasing the pH of the dispersion obtained by mixing said composition with water, to a value greater than or equal to 10 and preferably greater than or equal to 9; so that the water-resistance of a finish obtained from a slurry made by mixing of the dry composition with water to the correct consistency, with respect to the whole mix water/dry composition, said
10 water-resistance of the finish being measured according to the test ASTM D2486-06, is greater than, in an increased order of preference, 3000; 4000 ; 5000 ; 6000 ; 10 000 cycles;
- c) a specific adsorbent filler;
- d) optionally a load;
- 15 e) optionally water retentive agent;
- f) optionally a rheological agent;
- g) optionally a defoamer;
- h) optionally a biocide;
- i) optionally a pigment;
- 20 j) optionally fibers;
- k) optionally a mineral binder;
- l) optionally a flame retardant (or flame proof agent).

In one aspect, there is provided a redispersible dry composition of polymer finish comprising:

- a) a powdery organic binder included a polymer obtained by spray-drying or lyophilisation of a dispersion containing said polymer and a protective colloid;
- b) a deprotection agent chosen from the group consisting of glycol ethers, sodium triphosphate and mixes thereof; so that the water-resistance of a finish obtained from a slurry made by mixing of the dry composition with water to the correct consistency, with respect to the whole mix water/dry composition, said water-resistance of the finish being measured according to the test ASTM D2486-06, is greater than, in an increased order of preference, 3000; 4000 ; 5000 ; 6000 ; 10 000 cycles;
- c) a specific adsorbent filler whose oil absorption (OA), according to ISO 787/5 and in g / 100 g, is greater than or equal to, in an increased order of preference: 1; 5; 10; 20;
- d) optionally a load;
- e) optionally a water retentive agent;
- f) optionally a rheological agent;
- g) optionally a defoamer;
- h) optionally a biocide;
- i) optionally a pigment;
- j) optionally fibers;
- k) optionally a mineral binder; and
- l) optionally a flame retardant (or flame proof agent).

In another aspect, there is provided a wet formulation obtained by mixing water with the redispersible dry composition of polymer finish as disclosed herein, said formulation having a pH below 10, preferably below 9.

In another aspect, there is provided an application of the wet formulation as defined above, onto the surface of buildings or civil engineering works to produce coatings.

In another aspect, the disclosure provides buildings or civil engineering works or coatings applied thereon or finishes, obtained from the wet formulation as disclosed herein.

In another aspect, the disclosure provides finishes obtained from the wet formulation as defined above, the water resistance of said finish being measured according to the test ASTM D2486-06, being greater than, in an increased order of preference, 3000 ; 4000 ; 5000 ; 6000 ; 10 000 cycles; said finish being obtained from a slurry made by mixing of the dry composition with water to the correct consistency with respect to the whole mixed water / dry composition.

In a remarkable variant, the redispersible dry composition according to the invention, is characterized by a dry % mass ratio *specific filler c) *100 / polymer a)* that is greater or equal to, in an increasing preference order, 10, 20, 30, preferably greater or equal to 50, more preferably greater or equal to 150, and lower or equal to, in a decreasing preference order, 1200, 1000, 950, preferably lower or equal to 910, more preferably lower or equal to 850.

The thickness of the coating in the above mentioned test could vary from the one of ASTM D2486-06 norm. The thickness could increase if the granulometry of the largest load is

greater to the 180 microns requested in the ASTM D2486-06. The thickness of the coating should then correspond to the maximum diameter of the largest load.

The « correct consistency » could be for instance determined by comparison with the following products:

- 5
 - floor grout 5046 Elit from Parexlanko, mixed with water to a 27% (+/- 1%) water mix ratio (for floor joint fillers);
 - DPR Sandfine from the Parex company (for acrylic renders)
 - 451 Lankotoit from the Parexlanko company (for roof waterproofing membranes)

In a particular embodiment, the redispersible dry composition of polymer finish according to the invention, comprise:

- 5 a) a powdery organic binder included a polymer obtained by spray-drying or lyophilisation of a dispersion containing said polymer and a protective colloid;
- b) a deprotection agent;
- c) a specific adsorbent filler;
- d) a load;
- e) a water retentive agent;
- 10 f) optionally a rheological agent;
- g) optionally a defoamer;
- h) optionally a biocide;
- i) optionally a pigment.

15 This composition fulfils at least one of the above mentioned objects.

It is to the credit of the inventors to have designed a redispersible powder dry composition of plaster, mortar, rendering or finish, which is to address to the problem of redispersion in water of polymer-binded coatings or mass objects which are normally only water-resistant when resulting from alkaline aqueous slurries whose pH are greater than 10, especially
20 greater than or equal to 11, and preferably to 12. Now, $\text{pH} > 12$ are synonymous of formation of a powdery deposit on the surface of bodies, masonry units, etc., following a loss of water in open air (efflorescence).

In particular, the inventors succeeded in obtaining a dry composition which can be easily and rapidly transformed into a wet formulation, a slurry, the pH of which is below 10,
25 preferably below 9, by mixing with a liquid, e.g. water.

It is then possible to get a coating or a mass object from this slurry. Said coating is advantageously water-resistant and not subject to efflorescence.

Water-resistance is a very interesting property of the coatings or of the mass objects which are the results of $\text{pH} < 10$ slurries obtained from mixes of water and dry compositions
30 according to the invention. The water-resistance can be notably given by the wet scrub test and the drip test hereinafter defined.

This result has been obtained notably by incorporating into the dry composition, at least a deprotection agent b) and at least a specific adsorbent filler c).

Such a dry powder composition makes it also possible to overcome the drawbacks of water
35 in the known wet polymer finish formulations, namely strength to thermal shocks, biological instability and cost of transport.

Once redispersed in water, the dry composition forms a wet formulation with good properties of rheology, pumpability, workability, and durability.

And the hardened water-resistant and non-efflorescent coatings or the water-resistant and non-efflorescent joints or other mass objects obtained from this wet formulation have remarkable mechanical properties (hardness-cracking strength), and/or fireproofing and waterproofing properties, and/or decoration properties.

Definitions

According to the terminology of this text, the following non limitative definitions have to be taken into consideration:

- Every singular designates a plural and reciprocally.
- "*polymer*" refers to homopolymer or copolymer.
- "*finish*" refers to any product for the construction (interior or exterior), *e.g.* : coatings, joints, mass objects...
- The term "*redispersible*" or "*redispersibility*" used herein is taken to mean that the composition, if present in dry form, in particular as dry powder, is redispersible in aqueous media, in particular in water itself. This requirement of redispersibility is complied with by a re-obtained dispersion which is stable for preferably at least about 2 hours, in particular at least about 12 hours, and under ideal conditions more than 24 hours. In other words, there will not occur a significant precipitation. For the determination of such redispersibility the following method could preferably be employed: 50g of an essentially lump-free powder are mixed with 50g water in a beaker of a diameter of 8 cm and a maximum filling volume of 300 cm³. Stirring is performed with a propeller mixer (3 propellers and a standard diameter of 60 mm) with 1.000 rpm. The dispersion obtained is visually evaluated after 5, 10, 15, 30 minutes, and 1, 2, 3 hours respectively.
- "*deprotection agent*" refers to a compound or a mix of compounds which can wipe out at least one part of the protective effect of the protective colloid of the powdery organic binder a).
- "*cement*" is a mineral binder, free from any organic compound.
- "*cement-free*" means that the cement concentration is lower or equal to, in a increasing preference order and in % by weight of the composition, 5 ; 3 ; 1 ; 0,5 ; 10⁻¹; 10⁻³; 10⁻⁶.
- "*lime-free*" means that the lime concentration is lower or equal to, in a increasing preference order and in % by weight of the composition, 5 ; 3 ; 1 ; 0,5 ; 10⁻¹; 10⁻³; 10⁻⁶.
- "*granulometry*" corresponds to the dry sieving D₅₀.
- The granulometry of a "*powder*" is less or equal to 1 mm,

- The granulometry of a "*granulate*" is greater than 1 mm, preferably less or equal to 10 mm.
- "*Filler*" designates a fine and/or light mineral powdery loading material incorporated in the mortars, plasters, renderings or finishes, and the D_{50} of which is lower than 100 μm .
- "*Load*" designates a mineral powdery loading material incorporated in the mortars, plasters, renderings or finishes, and the D_{50} of which is greater than 100 μm .
- "*Acrylic*" refers to homopolymers or copolymers formed of monomers selected from the group of acrylate esters.

Detailed description of the invention

In a preferred embodiment, the powder dry composition according to the invention is cement-free and/or lime-free.

Powdery organic binder a):

The polymer of the binder a) is preferably chosen from the group comprising, preferably consisting of: acrylic, acrylic/acrylic, styrene/acrylic, styrene/butadiene, vinyl-acetate/ethylene, vinyl-acetate/versatate and vinyl-acetate/versatate/ethylene polymer; the acrylic, acrylic/acrylic, styrene/acrylic polymer being preferred.

In a variant, the binder a) can comprise a combination of different polymers.

The protective colloid of the binder a) is, for instance a polyvinyl alcohol, or a low molecular weight surface active compound, or taken from the group of salts of arylsulfonic acid formaldehyde condensates (U.S. Pat. No. 5,225,478).

Deprotection agent b)

Advantageously, the deprotection agent b) is chosen among those which are effective without increasing the pH of the dispersion obtained by mixing said composition with water, to a value greater than or equal to 10 and preferably greater than or equal to 9.

Regarding the quantitative aspect of the composition according to the invention, it is preferable that the dry % mass ratio *deprotection agent b)*100 / polymer a)* be greater or equal to, in an increasing preference order, 1, 8, 10, 15, 20, 30, preferably comprised between 15 and 25.

Actually, the deprotection agent b) is e.g. chosen in the group of products comprising, preferably consisting of glycol ethers, sodium triphosphate, the glycol ethers being more preferably chosen in the group of products comprising, preferably consisting of ethylene glycol monomethyl ether, ethylene glycol monoethyl ether, Ethylene glycol monopropyl

ether, ethylene glycol monoisopropyl ether(2-isopropoxy)ethanol, ethylene glycol monobutyl ether, ethylene glycol monophenyl ether, ethylene glycol monobenzyl ether, diethylene glycol monomethyl ether 2-(2-methoxyethoxy)ethanol, methyl carbitol, diethylene glycol monoethyl ether 2-(2-ethoxyethoxy)ethanol, carbitol cellosolve, 5 diethylene glycol mono-n-butyl ether, ethylene glycol dimethyl ether, ethylene glycol diethyl ether, ethylene glycol dibutyl ether, Ethylene glycol methyl ether acetate, ethylene glycol monethylether acetate, ethylene glycol monobutyl ether acetate tripropylene glycol n-butyl ether, dipropylene glycol n-butyl ether, triethylene glycol monobutyl etherpropylene glycol.

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Specific adsorbent filler c)

The specific adsorbent filler c) is "specific" with respect to its "adsorption capacity" and/or to its oil absorption (OA). In a particularly advantageous embodiment, the specific adsorbent filler c) has an oil absorption (OA) according to ISO 787/5 and in g / 100 g: OA 15 greater than or equal to, in an increased order of preference: 1; 5; 10; 20, and for example comprised between 5 and 10.

Said specific adsorbent filler c) is selected preferably among siliceous, calcareous or siliceous-calcareous powders and/or aluminosilicate powders, and more preferably in the calcareous or marble fillers, and, more preferably in the group consisting of the following 20 commercial products:

- beatite® : A carbonated load obtained from a "Pyrenean" calcite with a high chemical purity;
- Omyacarb®: A dry ground Calcium Carbonate with an intermediate and closely sized particle distribution;
- 25 - Mikhart® is a Calcium Carbonate and is produced by sieving or grinding the mineral Calcite.
- Omya TITAN® 200; Calcium Carbonate 97% Magnesium Carbonate 2% Median Diameter : 25 µm;
- CALCITEC PURO PH ® : Calcium Carbonate produced through careful selection 30 both in the mining and in the production stages, where dedicated lines are used to ensure high chemical purity characterized by a low content of heavy metals:

Load d)

Concerning the load d), it is advantageously chosen from the group of calcareous and/or 35 siliceous sands, and/or perlite and/or vermiculite.

Water retentive agent e)

The water retentive agent e) has the property to keep the water of mixing before the setting. The water is so trapped in the wet formulation paste which improves its bond. To
5 some extent, the water is less absorbed by the support. Salting out on the surface is limited and evaporation is reduced.

The water retentive agent e) is preferably chosen in the group comprising: modified cellulose ethers and/or starches ethers and/or guar ether and their mixes, more preferably
10 consisting of: methylcelluloses, methylhydroxypropylcelluloses, methylhydroxyethylcelluloses and their mixes.

The possible rheological agent f) (also named a "thickener") is preferably chosen in the group comprising, more preferably consisting of : clays, starch ethers, cellulose ethers and/or gums (e.g. Welan guar xanthane, succinoglycans), modified polysaccharides -
15 preferably among modified starch ethers-, polyvinyl alcohol, polyacrylamides, clays, sepiolites, bentonites, and their mixes, and more preferably chosen in the group of clays, bentonite, montmorillonite.

The possible defoamer g) is preferably chosen in the group comprising, more preferably
20 consisting of: polyether polyols and mixes thereof.

The possible biocide h) is preferably chosen in the group comprising, more preferably consisting of: mineral oxides like zinc oxide and mixes thereof.

25 The possible pigment i) is preferably chosen in the group comprising, more preferably consisting of: TiO_2 , iron oxide and mixes thereof.

The possible fibers j) are preferably chosen in the group comprising, more preferably consisting of fibers of polyamid, polyacrylonitrile, polyacrylate, cellulose, polypropylene,
30 glass, sisal, or hemp and their mixes.

The possible mineral binder k) is preferably chosen in the group comprising, more preferably consisting of: portland cement, calcium aluminate cement, calcium sulphate and their mixes.
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The possible flame retardant (or flame proof agent), which makes it possible to increase the fire resistance and/or to shrink the speed of flame spreading of the composition is preferably chosen in the group comprising, more preferably consisting of:

- minerals preferably aluminium hydroxide [Al(OH)₃, ATH], magnesium hydroxide MDH, hydromagnesite, hydrates, red phosphorus, and boron compounds, preferably borates,
- organohalogen compounds, preferably organochlorines and more preferably such as chlorendic acid derivatives and chlorinated paraffins; organobromines such as decabromodiphenyl ether (decaBDE), decabromodiphenyl ethane,
- polymeric brominated compounds preferably brominated polystyrenes, brominated carbonate oligomers (BCO's), brominated epoxy oligomers (BEO's), tetrabromophthalic anhydride, Tetrabromobisphenol A (TBBPA) and hexabromocyclododecane (HBCD).
- antimony preferably pentoxide and sodium antimonite
- organophosphorus compounds preferably organophosphate, TPP, RDP, BPADP, tri-o-cresyl phosphate,
- phosphonates preferably DMMP and phosphinates.
- chlorophosphates like TMCP and TDCP.

The composition according to the invention can also comprise some optional functional ingredients like

- air-entraining agents (surfactants e.g. natural resins, sulfated or sulfonated compounds, synthetic detergents, organic fatty acids and their mixes, preferably among the lignosulfonates, the basic soaps of fatty acids and their mixes, and, more preferably in the group comprising the sulfonate olefins, the sodium lauryl sulfate de sodium and their mixes;
- accelerators (calcium salts, carbonates, preferably lithium or sodium and their mixes);
- retarders (tartric acid and its salts: sodium or potassium salts, citric acid and its salts: sodium (trisodic citrate) and their mixes;

As regards the quantitative data, the dry powder redispersible composition according to the invention can be the following, in percent by weight and in an increased preference order:

- $1 \leq \text{binder a)} \leq 40$; $1 \leq \text{binder a)} \leq 30$; $5 \leq \text{a)} \leq 25$; $6 \leq \text{a)} \leq 20$; $8 \leq \text{a)} \leq 15$;
- $0,1 \leq \text{deprotection agent b)} \leq 10$; $0,5 \leq \text{b)} \leq 8$; $0,8 \leq \text{b)} \leq 5$; $1 \leq \text{b)} \leq 4$;
- $1 \leq \text{specific adsorbent filler c)} \leq 85$; $1 \leq \text{specific adsorbent filler c)} \leq 75$; $5 \leq \text{c)} \leq 70$; $15 \leq \text{c)} \leq 60$;

- $25 \leq c) \leq 50$;
- $10 \leq \text{load d}) \leq 90$; $20 \leq d) \leq 80$; $30 \leq d) \leq 70$; $40 \leq d) \leq 60$;
 - $0,001 \leq \text{water retentive agent e}) \leq 5$; $0,01 \leq e) \leq 3$; $0,05 \leq e) \leq 1$;
 $0,08 \leq e) \leq 1$;
 - 5 ○ $0,001 \leq \text{rheological agent f}) \leq 5$; $0,05 \leq f) \leq 3$;
 $0,08 \leq f) \leq 3$; $0,1 \leq f) \leq 2$
 - $0,01 \leq \text{defoamer g}) \leq 5$; $0,05 \leq g) \leq 3$; $0,1 \leq g) \leq 2$;
 $0,2 \leq g) \leq 1$;
 - 10 ○ $0,001 \leq \text{biocide h}) \leq 5$; $0,01 \leq h) \leq 3$; $0,05 \leq h) \leq 1$;
 $0,08 \leq h) \leq 1$
 - $0,01 \leq \text{pigment i}) \leq 9$; $0,05 \leq i) \leq 3$; $0,1 \leq i) \leq 2$;
 $0,2 \leq i) \leq 1$

15 The present invention also concerns a wet formulation obtained by mixing water with the redispersible dry composition of polymer finish as above defined, said formulation being preferably plasters, renderings, finishes, grouts, waterproofing coatings or mortars. The mixing is done by any appropriate device known in the art. The mixing time is e.g. 1 to 30 min, or even 3 to 10 min.

20 The water content of such a wet formulation is preferably comprised between, increasing order of preference and in % by weight of the wet formulation, 5-50 ; 10-40 ; 15-25.

Another object of the invention is the application of the wet formulation as above defined onto the surface of buildings or civil engineering works to produce coatings.

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Another object of the invention is the buildings or civil engineering works or coatings applied thereon or finishes, obtained from the wet formulation as above defined.

30 The coatings and the mass works or objects obtained from the redispersible powder polymer composition according to the invention have good application properties.

In particular, the coatings according to the invention produce good results in the usual assessment tests, namely: impact resistance, wet scrub resistance 7 day cure, mandrel bend test 7 day cure, tinting strength, QUV test (accelerated weathering) and drip-test measuring the water-resistance in terms of redispersibility, hardness, colour stability, abrasion
 35 resistance and texture appearance.

EXAMPLES

-I- Raw materials

- 5 a) powdery organic binder:
- (a.1) redispersible powder acrylic polymer commercialized by ELOTEX under the name WR8600
- (a.2) redispersible powder styrene/acrylic polymer commercialized by BASF under the name ACRONAL[®] S695P
- 10 (a.3) redispersible powder acrylic polymer commercialized by WACKER[®] under the name Vinnapas[®]1540 N
- (a.4) redispersible powder Vinylacetate / Ethylene polymer commercialized by WACKER[®] under the name Vinnapas[®]5010 N
- 15 b) Deprotection agent:
- Metolat[®] 872P commercialized by Münzig: Blend of aliphatic alcohols and glycols (notably triethylene glycol monobutyl ether) with an inorganic carrier: content of triethylene glycol monobutyl ether in the powder : 25 to 50%.
- 20 c) Specific adsorbent filler:
- (c.1) calcareous filler commercialized by Omya[®] under the name Beatite[®] 30
- (c.2) calcareous filler commercialized by Omya[®] under the name Hydrocarb[®] -OG
- 25 d) a load:
- (d.1) calcareous sand commercialized by la provençale[®] under the name Calgar[®]
- 30 (d.2) siliceous sand commercialized by Sibelco France under the name N14
- (d.3) calcareous sand commercialized by Omya[®] under the name Durcal[®] 130
- 35 e) a water-retentive agent:
- cellulosic ether: commercialized by Dow[®] chemicals under the name Methocel[®]

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- f) rheological agent:
- (f.1) sepiolite commercialized by TOLSA[®] under the name Pangel[®] S9
 - (f.2) silicate mineral commercialized by Rio Tinto Minerals[®] under the name Talc de Luzenac
- 10
- g) defoamer:
- (g.1) polyglycols commercialized by MUNZIG under the name Agitan P823
 - (g.2) polyethers polyols commercialized by KERNEOS[®] under the name Peramin[®] defoam 50 PE
- 15
- h) biocide:
- zinc oxyde commercialized by Troy[®] under the name Troysan[®] CP 20
- 20
- i) pigment:
- (i.1) TiO₂, commercialized by TWT under the name Titanpol R03
 - (i.2) Iron oxide commercialized by TWT under the name Noir 318
 - (i.3) Sodium alumino-silicate commercialized by holidays pigments under the name Bleu outremer 03
- 25
- j) fibers:
- (j1) cellulosic fiber commercialized by JRS[®] under the name Arbocel B00
 - (j2) polyacrylonitrile fibers commercialized by STW[®] under the name PAC243/125
- 30
- k) mineral binders
- (k1) Calcium Aluminate Cement commercialized by Kerneos[®] under the name Ternal RG
 - (k2) Calcium Sulfate binder commercialized by Maxit[®] under the name Anhydrite micronisée Micro A
- 35
- l) and/or a flame retardant (or flame proof agent):
- aluminium hydroxide commercialized by Sibelco[®] under the name portaflame SG 200

-II- Method

The redispersible powder compositions of the examples are prepared as follows:

- Each raw material is weighed according to the proportions of the compositions given below in the point III, then introduced into a classical powder mixer. The mixing time varies between 45 sec. and 10 minutes depending on the composition and on the mixer's efficacy.

10 **-III- The compositions****III.1 Renders, plasters**

In % by weight

15

Example Formula 1

d.1	50
c.1	32
a1	11
i1	3
b	2,2
f1	1
g1	0,4
e	0,1
h	0,1
TOTAL	100

Example Formula 2

d.2	50
c.1	34,8
a.1	12
b	3
e	0,2
TOTAL	100

Example Formula 3

c.1	44,6
d.2	29
d.1	12
a2	10
b	2
i.1	1,25
f1	0,5
g2	0,5

e	0,15
TOTAL	100

Example Formula 4

c.1	44,13
d.2	30
d.1	12
a.1	9
b	4,8
e	0,07
TOTAL	100

Example Formula 5

c.1	63,4
a.3	25
i.1	4
b	3,5
f.1	3
g.2	0,7
e	0,4
TOTAL	100

Example Formula 6

d.1	50
c.1	17,2
l	15
a.1	11
i.1	3
b	2,2
f.1	1
g.1	0,4
e	0,1
h	0,1
TOTAL	100

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III.2 Grouts**Example Formula 7**

c.1	77,375
a.1	12
f.2	5
b	2,4
i.1	2
j.1	1

15

i.2	0,2
i.3	0,025
TOTAL	100

Example Formula 8

c.1	80
a.1	12
f.2	5
i.1	2
j.1	1
b	0
TOTAL	100

Example Formula 9

c.2	45
c.1	40,6
a.1	12
b	2,4
TOTAL	100

5

Example Formula 10

c.2	76
a.1	20
b	4
TOTAL	100

Example Formula 11

d.3	60
c.1	32
a.4	6
b	1
j2	1
TOTAL	100

10

Example Formula 12

d.3	60
c.1	34
a.4	4
b	1
j2	1
TOTAL	100

Example Formula 13

d.3	60
c.1	31
a.4	8
j.2	1
b	0
TOTAL	100

III.3 Waterproofing

Example Formula 14

c.1	57,5
a.1	40
b	2
f.1	0,5
TOTAL	100

5

Example Formula 15

c.1	75,5
a.1	20
b	4
f.1	0,5
TOTAL	100

Example Formula 16

c.1	77,5
a.1	20
b	2
f.1	0,5
TOTAL	100

10 **III.4 Formula containing mineral binders**

Example Formula 17

d.1	50
c.1	31,2
a.1	11
i.1	3
b	2,2
k.1	1
f.1	1
g	0,4
e	0,1
h	0,1
TOTAL	100

Example Formula 18

d.3	60
c.1	27
a.4	6
k.2	5
b	1
j2	1
TOTAL	100

-IV- Application properties

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IV.1 Preparation of the samples

The final product is got by mixing of the powder with some water or inversely, till the obtaining of a homogeneous paste. The water contents for the above mentioned formulas according to the examples are:

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Formula 1: 19% +- 1%	Formula 7: 20%+- 1%	Formula 13: 25%+-1%
Formula 2: 16% +-1%	Formula 8: 16%+-1%	Formula 14: 30%+- 1%
Formula 3: 17% +-1%	Formula 9: 24%+-1%	Formula 15: 19%+-1%
Formula 4: 16%+-1%	Formula 10: 31%+-1%	Formula 16: 16%+-1%
Formula 5: 21%+-1%	Formula 11: 25%+-1%	Formula 17: 30%+- 1%
Formula 6: 20% +- 1%	Formula 12: 25%+-1%	Formula 18: 22%+- 1%

IV.2 The tests

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a) DRIP TEST

• Goal:

This test aims at determining the water-resistance of the compositions according to the invention, in terms of redispersibility, hardness, colour stability, abrasion resistance and texture appearance.

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In this respect, a “*driptest*” device was set up to simulate the impact of a rain shortly after application. Low mineral content water will hence be used.

Two commercial products (acrylic renders in pail) which stand for our references, have undergone the test first, to determine test duration and the minimum curing time required before running the experiment. Then, those data may differ accordingly with the nature of the products tested.

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- Drip test principle for acrylic renders:

Deposits will undergo a constant drip of deionised water, after the determined curing times (4 to 8 hours for acrylic renders) over 30 and 60 minutes.

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Flow: 9-12 ml/min

Waterfall height: around 23 cm

Device and settings: Watson-Marlow Limited, 313 Ambient 5C to 40C/41F to 104F / On time secs = 1.5 / Off time secs = 2.0

- The following points will then be checked out:

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- Abrasion resistance: if the impact of water drops damages the surface (erosion), abrasion resistance under water is considered as poor (marked as "Yes").

- Colour stability: after rain, the sample is bound to dry again without any spots or whitening appearing on the surface.

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- Recovery: Sample should recover its hardness and texture appearance after re-drying.

- Redispersibility: right after the driptest, the deposit is checked by scrubbing the surface with a finger (light pressure). As the samples are tinted, redispersibility of the material is determined by the presence of coloured water on the gloves (marked as "Yes").

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b) WET SCRUB RESISTANCE 7 DAYS CURE

Test valuate washability and wet abrasion resistance of a material. The tests reciprocate a wet abrasion force linear over the sample: according Standard ASTM D2486-06 Standard Test Methods for water Scrub Resistance of Wall Paints. The thickness of the finish in the above mentioned test could vary from the one of ASTM D2486-06 norm. The thickness could increase if the granulometry of the largest load is greater to the 180 microns requested in the ASTM D2486-06. The thickness of the finish should then correspond to the maximum diameter of the largest load.

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c) MANDREL BEND TEST 7 DAYS CURE

The Mandrel Bend Test covers the determination of the resistance to cracking (flexibility) of an organic coatings applied on substrates of sheet metal. The coating materials under test are applied at uniform thickness to panels of sheet metal. After drying the coated panels are bent over a mandrel and the resistance to cracking of the coating is determined. The noted value is the diameter of the cylinder for which there is no crack.

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d) TINTING STRENGTH

Tinting strength is a test which makes it possible to check the colouring capacity of pigments in a given composition. Identical quantities of pigments are introduced in a control composition (here a pasty acrylic finish) and in the sample according to the invention (to be tested). The two-coloured compositions are applied onto a standard support and dried. The tinting strength is measured by a spectrophotometer to determine the cie Lab* and the ΔE (difference of tint between) according to the standard ASTM E-308. If ΔE is less than 1.5, the tints are considered as identical.

e) QUV ACCELERATED WEATHERING

The QUV test method is a cyclic ultraviolet weathering procedure. The particular test equipment uses a UV energy source that consists of eight 40-W fluorescent lamps with an emission range of 280 to 350 nm, with continuous peak output at 310 NM. The fluorescent lamps emit UV radiation that emulates the intensity of mid-day June sunlight in Florida. During the exposure tests, oxygenated water is applied to the test samples by condensation, rather than by spray. The water supply is heated in a vented tank below the test racks and lamps. To control the cyclic tests, the QUV is equipped with a variable interval timer that alternates the UV and the condensation at 43° C followed by 8 hours UV radiation at 60° C and high humidity.

IV.3 Renders, plasters results

TESTING PARAMETERS	PAREX DPR SAND FINE	FORMULA 1
Drip test 24-HRS	No erosion colour stability	No erosion colour stability
DRIP TEST (AFTER 4 HRS)	No erosion colour stability	No Erosion colour stability
SCRUB RESISTANCE 7 DAYS CURE	8,000 + CYCLES	8,000 + CYCLES
MANDREL BEND TEST 7 DAYS CURE	3/16" inch	1/4" inch

TINTING STRENGTH	Same colour	Same colour
QUV ACCELERATED WEATHERING		
500 HRS	Colour stability	Colour stability
2,000 HRS	Colour stability	Colour stability

PAREX DPR SAND FINE = trade composition of pasty acrylic finish commercialized in the USA by the PAREX[®] company = control.

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IV.4 Grouts results

TESTING PARAMETERS	Scrub Resistance	Scrub Resistance
	24 Hours Cure	7 Days Cure
Formula 7	>10000 cycles	>10000 cycles
Formula 8	2300 cycles	2500 cycles
Formula 9	>10000 cycles	>10000 cycles
Formula 10	>10000 cycles	>10000 cycles
Formula 11	-	>10000 cycles
Formula 12	-	4500 cycles
Formula 13	-	600 cycles

IV.5 Waterproofing results

TESTING PARAMETERS	Formula 14	Formula 15	Formula 16
Scrub Resistance 24 Hours Cure	>17000 cycles	>17000 cycles	>17000 cycles
Scrub Resistance 7 Days Cure	>17000 cycles	>17000 cycles	>17000 cycles

IV.6 Mineral binders results

TESTING PARAMETERS	pH value
Formula 17	9,6
Formula 18	6,9

CLAIMS

1. Redispersible dry composition of polymer finish comprising:
 - 5 a) a powdery organic binder included a polymer obtained by spray-drying or lyophilisation of a dispersion containing said polymer and a protective colloid;
 - b) a deprotection agent chosen from the group consisting of glycol ethers, sodium triphosphate and mixes thereof; so that the water-
 10 resistance of a finish obtained from a slurry made by mixing of the dry composition with water to the correct consistency, with respect to the whole mix water/dry composition, said water-resistance of the finish being measured according to the test ASTM D2486-06, is greater than, in an increased order of preference, 3000; 4000 ; 5000 ;
 15 6000 ; 10 000 cycles;
 - c) a specific adsorbent filler whose oil absorption (OA), according to ISO 787/5 and in g / 100 g, is greater than or equal to, in an increased order of preference: 1; 5; 10; 20;
 - d) optionally a load;
 - 20 e) optionally a water retentive agent;
 - f) optionally a rheological agent;
 - g) optionally a defoamer;
 - h) optionally a biocide;
 - i) optionally a pigment;
 - 25 j) optionally fibers;
 - k) optionally a mineral binder; and
 - l) optionally a flame retardant (or flame proof agent).

2. Redispersible dry composition according to claim 1 wherein the dry % mass ratio
 30 *specific filler c) *100 / polymer a)* that is greater or equal to, in an increasing preference order, 10, 20, 30, preferably greater or equal to 50, more preferably greater or equal to 150, and lower or equal to, in a decreasing preference order, 1200, 1000, 950, preferably lower or equal to 910, more preferably lower or equal to 850.

3. Redispersible dry composition of polymer finish according to claim 1 comprising:
- a) the powdery organic binder including a polymer obtained by spray-drying or lyophilisation of a dispersion containing said polymer and a protective colloid;
 - b) the deprotection agent;
 - c) the specific adsorbent filler;
 - d) the load;
 - e) the water retentive agent;
 - f) optionally the rheological agent;
 - g) optionally the defoamer;
 - h) optionally the biocide; and
 - i) optionally the pigment.
4. A composition according to claim 1 which is cement-free and/or lime-free.
5. A composition according to claim 1 or 2, wherein the binder a) is a homopolymer or copolymer chosen from the group comprising, preferably consisting of: acrylics, styrene/acrylic, styrene/butadiene, vinyl-acetate/ethylene, vinyl-acetate/versatate and vinyl-acetate/versatate/ethylene polymer, acrylic /acrylic copolymer being preferred.
6. A composition according to claim 1, wherein the deprotection agent b) is chosen in the group consisting of ethylene glycol monomethyl ether, ethylene glycol monoethyl ether, ethylene glycol monopropyl ether, ethylene glycol monoisopropyl ether, (2-isopropoxy)ethanol, ethylene glycol monobutyl ether, ethylene glycol monophenyl ether, ethylene glycol monobenzyl ether, diethylene glycol monomethyl ether 2-(2-methoxyethoxy)ethanol, glycol ether with the following physical characteristics:

<u>Molecular weight (g/mol)</u>		<u>120.1</u>
<u>Boiling point @ 760 mmHg, 1.01 bar</u>	<u>381°F</u>	<u>194°C</u>
<u>Flash point</u>	<u>205°F</u>	<u>96°C</u>
<u>Freezing point</u>	<u>-92°F</u>	<u>-69°C</u>
<u>Vapor pressure @ 20°C — extrapolated</u>		<u>0.19 mmHg</u> <u>0.25 mbar</u>
<u>Specific gravity (25/25°C)</u>		<u>1.020</u>
<u>Density @ 20°C</u>	<u>8.53 lb/gal</u>	<u>1.022 g/cm³</u>
<u> @ 25°C</u>	<u>8.49 lb/gal</u>	<u>1.017 g/cm³</u>
<u>Viscosity (cP or mPa·s @ 25°C)</u>		<u>3.5</u>
<u>Surface tension (dynes/cm @ 25°C)</u>		<u>24.6</u>
<u>Specific heat (J/g°C @ 25°C)</u>		<u>2.30</u>
<u>Heat of vaporization (J/g) at normal boiling point</u>		<u>369</u>
<u>Net heat of combustion (kJ/g) — predicted @ 25°C</u>		<u>23.6</u>
<u>Autoignition temperature</u>	<u>464°F</u>	<u>240°C</u>
<u>Evaporation rate</u>	<u>(n-butyl acetate = 1.0)</u> <u>(diethyl ether = 1.0)</u>	<u>0.019</u> <u>628</u>
<u>Solubility, g/100 g @ 25°C</u>		<u>-</u>
<u> Solvent in water</u>		<u>-</u>
<u> Water in solvent</u>		<u>-</u>
<u>Hansen solubility parameters (J/cm³)^{1/2}</u>		<u>16.2</u>
<u> d (Dispersion)</u>		<u>7.8</u>
<u> p (Polar)</u>		<u>12.6</u>
<u> h (Hydrogen bonding)</u>		<u>-</u>
<u>Flammable limits (vol. % in air)</u>		<u>1.38</u>
<u> Lower</u>		<u>22.70</u>
<u> Upper</u>		<u>-</u>

diethylene glycol monoethyl ether 2-(2-ethoxyethoxy)ethanol, diethylene glycol mono-n-butyl ether, ethylene glycol dimethyl ether, ethylene glycol diethyl ether, ethylene glycol dibutyl ether, ethylene glycol methyl ether acetate, ethylene glycol monethylether acetate, ethylene glycol monobutyl ether acetate tripropylene glycol n-butyl ether, dipropylene glycol n-butyl ether, and triethylene glycol monobutyl etherpropylene glycol.

7. A composition according to any of the preceding claims, wherein the specific adsorbent filler c) is selected from aluminosilicates, calcareous or marble fillers and mixes thereof.
8. A composition according to any of the preceding claims, wherein the dry % mass ratio *deprotection agent b)*100 /polymer a)* is greater or equal to, in an increasing preference order, 1, 8, 10, 15, 20, 30, preferably comprised between 15 and 25.
9. A composition according to any of the preceding claims, wherein the load d) is chosen from the group of calcareous and/or siliceous sands and/or vermiculite and/or perlite.
10. A wet formulation obtained by mixing water with the redispersible dry composition of polymer finish according to any of the preceding claims, said formulation having a pH below 10, preferably below 9.
11. Application of the wet formulation according to claim 10, onto the surface of buildings or civil engineering works to produce coatings.
12. Buildings or civil engineering works or coatings applied thereon or finishes, obtained from the wet formulation according to claim 10.
13. Finishes obtained from the wet formulation according to claim 10, the water resistance of said finish being measured according to the test ASTM D2486-06, being greater than, in an increased order of preference, 3000 ; 4000 ; 5000 ; 6000 ; 10 000 cycles; said finish being obtained from a slurry made by mixing of the dry composition with water to the correct consistency with respect to the whole mixed water / dry composition.

14. A composition according to claim 1, wherein the deprotection agent b) is chosen in the group consisting of ethylene glycol monomethyl ether, ethylene glycol monoethyl ether, ethylene glycol monopropyl ether, ethylene glycol monoisopropyl ether, (2-isopropoxy)ethanol, ethylene glycol monobutyl ether, ethylene glycol monophenyl ether, ethylene glycol monobenzyl ether, diethylene glycol monomethyl ether 2-(2-methoxyethoxy)ethanol, diethylene glycol monoethyl ether 2-(2-ethoxyethoxy)ethanol, diethylene glycol mono-n-butyl ether, ethylene glycol dimethyl ether, ethylene glycol diethyl ether, ethylene glycol dibutyl ether, ethylene glycol methyl ether acetate, ethylene glycol monethylether acetate, ethylene glycol monobutyl ether acetate tripropylene glycol n-butyl ether, dipropylene glycol n-butyl ether, and triethylene glycol monobutyl etherpropylene glycol.