

PATENT SPECIFICATION

(11) 1 408 083

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- (21) Application No. 2124/73 (22) Filed 15 Jan. 1973
 (31) Convention Application No. 6051/72 (32) Filed 13 Jan. 1972 in
 (33) Japan (JA)
 (44) Complete Specification published 1 Oct. 1975
 (51) INT CL² B05D 5/00
 (52) Index at acceptance
 B2E 19Y 206 20Y 219 228 327 372 382 383 38Y 412 413
 41Y 44Y 461 466 478 498 519 65Y 671 677 708
 71Y 806
 C1H 120 610 620 621 717 718 724 730 739 745 757 768
 772 783 812



(54) DECORATIVE LAYER

(71) We, THE BONNTILE CO. LTD., a Japanese Company of No. 4, Kyobashi 3-chome, Chuo-ku, Tokyo, Japan; and DAI NIPPON TORYO CO. LTD., a Japanese Company of 38, Nishino Shimonochi, Konohana-ku, Osaka-shi, Osaka-fu, Japan, 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:—

This invention relates to a method for preparing a decorative layer especially on the interior or exterior walls or the ceilings of a building.

It is known that a cement type mortar or an organic mortar, such as vinylacetate resin, acrylic ester resin etc., can be air blasted or sprayed on to the inner or outer walls of a building, which walls may be made of concrete, mortar, slate, foamed concrete or metal plate. The coating of walls in this manner is widely carried out for improving the water-proofing properties and the durability of the wall and for imparting an attractive pattern. However, the conventional decorative layer has certain disadvantages. For example, when the decorative layer is prepared by using a cement-type mortar, the surface of the base must be undercoated with a synthetic resin emulsion to improve the adhesion of the mortar to the base. Also cracks are usually formed in the surface of the layer when it hardens. When the decorated layer is hardened in summer or in a dry atmosphere, it is necessary to use an under-treatment consisting of sprinkling the surface of the base with for providing adequate moisture or to form a moisture vaporization-preventing layer, so that the layer does not dry

out prematurely before proper curing and hardening has taken place.

When the decorative layer is prepared by using an organic mortar, these can often be an undesirable delay in the hardening of the layer in an environment of high humidity and usually there occurs difficulty in the pressing or trowelling operation which is used for forming a decorative pattern on the layer.

According to the invention there is provided a method of preparing a decorative layer which comprises spray-coating a mortar composition containing 3—50 wt% of an epoxy resin and an epoxy hardener, 5—70 wt% of cement, 5—70 wt% of aggregate powder and 5—30 wt% of water onto the surface of a base to form a layer having an uneven surface, allowing the coating to achieve a semi-hardened condition and then pressing the surface of the layer to flatten the tops of the projecting parts.

The invention at least in the preferred embodiment, thus provides a relatively simple method for preparing a decorative layer having a special surface appearance which is different from the conventional pattern and which has good durability, good adhesion to the base and which involves a relatively simple operation.

The invention will be further described in the following with reference to the accompanying drawings, in which:—

Figure 1 is a sectional view of a spray-coated decorative layer,

Figure 2 is a sectional view of the decorative layer of Fig. 1 after the tops of the projecting parts have been pressed with a roller or trowel while the layer was still in the semi-hardened condition; and

Figure 3 is a perspective view of a roller used for the pressing operation.

In the method of this invention, the epoxy resin is usually used in the form of an emulsion, the emulsion, from the view point of stability, is preferably an o/w (oil in water) type emulsion containing 40—60 wt% of the epoxy resin.

The epoxy resin is preferably a liquid epoxy resin prepared from bisphenol-A and epichlorohydrin, which is uniformly emulsified using a mainly nonionic surfactant or another type of emulsifier.

Typical epoxy resins which can be used in this invention are epoxy resins having at least two terminal epoxy groups and which are liquid at room temperature, such as bisphenol A-epichlorohydrin types, side-chain types, alicyclic types, polymerized aliphatic ester types, novolak types, butyl glycidyl ether types, polyalkylene types, glyceryl triepoxide types, and alkylphenol type epoxy resins.

Suitable liquid epoxy resins having at least two terminal epoxy groups are epoxidized glycerin dialiphatic ester, 1,4-bis (2,3-epoxy-propoxy) benzene, 1,3'-bis (2,3-epoxy-propoxy) benzene, 4,4'-bis (2,3-epoxy-propoxy) diphenylether, 1,8-bis (2,3-epoxy-propoxy) octane, 1,4-bis (2,3-epoxy-propoxy) cyclohexane, 4,4 - bis (2 - hydroxy - 3,4' - epoxy - butoxy) diphenyldimethyl methane, 1,3-bis (4,5-epoxy-pentoxo) 5-chlorobenzene, 1,4 - bis (3,4 - epoxy - butoxy) - 2 - chloro-cyclohexane, 1,3 - bis (2 - hydroxy - 3,4-epoxy - butoxybenzene and 1,4 - bis (2-hydroxy-4,5-epoxy-pentoxo) benzene.

It is especially preferable to use an epoxy polyether of a polyhydric phenol.

It is possible to use a mixture of 70% by weight or more of a liquid epoxy resin and 30% by weight or less of a solid epoxy resin which can be soluble in the liquid epoxy resin.

As stated before, epoxy resin is usually emulsified with a surfactant to form an o/w type emulsion.

The surfactant can be an anionic surfactant such as soap, alkyl sulfate, alkyl sulfonate, alkylaryl sulfonate, and sulfosuccinate, or a nonionic surfactant, such as a polyoxyethylene ether type, a polyoxyethylene ester type, a sorbitan ester type and a monglyceride type nonionic surfactant.

When the epoxy resin emulsion is admixed with the cement, the epoxy hardner and the aggregate and the composition is frozen and then melted, the epoxy resin still has film formability.

The epoxy hardener can be a conventional hardener used for conventional epoxy emulsions such as a polyamine, an amine adduct or a polyamide.

It is especially preferable to use as the

epoxy hardener a polyamide having the formula



wherein n represents an integer and R represents a C₁₋₄ alkyl group.

The polyamide can be prepared by the condensation of a polycarboxylic acid and a diamine.

When the said polyamide is used as the epoxy hardener, the flexibility, adhesiveness and chemical resistance of the product are improved compared with the product employing an amine as the epoxy hardener.

The epoxy resin in the form of an emulsion is uniformly mixed with the cement, the aggregate powder, a coloring agent and other additives, if any.

It is not advantageous to use an epoxy resin solution prepared by dissolving the epoxy resin in an organic solvent, because the epoxy resin solution cannot then be uniformly mixed with the cement and the aggregates the resulting operational time is too short, the adhesion is too low because of the low moisture content of the composition, there is a fire risk and the solvent may be toxic. The epoxy resin imparts higher adhesivity, alkali-resistance, chemical-resistance, water-resistance, weatherbility and lower contraction in comparison with, for example, vinylacetate type resins and acrylic type ester resins, so that it is advantageous to use the method of the invention to form a decorative layer on an exterior wall.

The cement used in the method of this invention is preferably a hydraulic cement such as white cement, Portland cement, alumina cement, or a mixture thereof.

The aggregate powder can be, for example, silica sand, volcanic ash, diatomaceous earth or sludge. The aggregate powder is usually a fine powder of 30 mesh (ASTM Standard) or smaller so as to impart suitable viscosity to the mortar composition, suitable sprayability through the nozzle and suitable pressability by the roller or the trowel.

It is preferable, from a view point of density and strength of the decorative layer, to use an aggregate powder having more than about 60 wt% of fine powder of 50—200 mesh.

In the mortar composition, if desirable, other additives, such as a pigment for imparting a desirable color and a hardening accelerator, such as calcium chloride, can be added.

The reason why the amount of the cement is limited to 5—70 wt% is as follows.

When the amount cement is less than 5 wt%, the adhesion of the composition deteriorates at low temperatures, and when the composition is pressed with a roller or a trowel, the mortar composition adheres to

the roller or the trowel so that it is difficult to obtain a uniform pattern.

When the amount of the cement is higher than 70 wt%, the resin content is relatively decreased, so that the adhesion is decreased. Because of these reasons, the optimum amount of cement is 5—70 wt%, and the results of this invention can be attained within the said range.

The reason why the amount of the epoxy resin and the epoxy hardener is limited to 3—50 wt% is as follows.

When the amount is less than 3 wt%, the adhesion is inferior and the pressability is decreased. On the other hand, when the amount is higher than 50 wt%, the resin content is increased so as to improve the adhesion, but the composition is stiffer and is difficult to press with a roller or a trowel because of the high tackiness, and also the cost of the composition is increased because of high epoxy resin content.

In the epoxy resin emulsion, it is usual to mix the epoxy resin and the epoxy hardener in the ratio of 1:0.5—1.5 by weight, preferably 1:0.7—1.2 by weight. If necessary, 0—20 wt% of other additives, such as pigments, tackifiers, preservatives, and emulsifiers can be added. The mortar composition containing said components is admixed with 5—30 wt%, preferably 15—25 wt%, of water and is kneaded well to prepare a mortar composition having suitable viscosity for spray coating i.e. 100—500 poise at 25°C. It is preferable initially to mix the cement and the epoxy resin hardener separately from the paste of the epoxy resin emulsion and the aggregate powder.

The above viscosity range is suitable for wetting the surface of the aggregate powder and for imparting high adhesion to the base and for forming an uneven surface on spraying. When the viscosity is too low, the fluidity is too high, so that the surface of the coating does not take on such a marked uneven pattern.

On the other hand, when the viscosity is too high the adhesion is too low so that the amount of mortar composition which falls away from the surface during spray coating increases.

It is especially suitable to use the following ranges for the composition: white cement 25—55 wt%, aggregate 15—30 wt%, water 2.6—18 wt%, epoxy resin 4—12 wt% and epoxy hardener 2—18 wt%.

The mortar composition can be spray-coated by a spray-gun onto the base surface. It is most preferable to spray-coat the mortar composition by a gun having a diameter of 4—12 mm at an air pressure of 2—8 kg/cm² so as to form suitable concavo and convex shapes. The sprayed mortar composition reaches the base surface in granule form so as to form a decorative surface having a concavo-convex,

i.e. uneven, pattern whose sectional view is shown in Figure 1.

It is especially preferable to use an undercoat of the mortar composition on the base surface by spraying the composition at a pressure of 4—8 kg/cm² as a first coating step, and then to coat with rough particles of the mortar composition so as to form the uneven or concavo-convex surface by spraying at a pressure of 2—5 kg/cm² as a second coating step.

In Figure 1, the reference numeral 1 designates a base surface, 2 designates a decorative coated layer 3 designates aggregate powder within the decorative layer and 4 designates projecting parts of the decorative layer.

After curing and semi-hardening for about 0.5—6 hours, in order to obtain the characteristic surface pattern of this invention the projecting parts of the decorative layer are pressed with a roller or a trowel so as to form projecting parts 3 having flat tops. The pressing operation by the roller or the trowel is performed during the time the decorative layer of the mortar composition is not finally hardened and has an amount of plasticity. In order to form the flat top of the convex parts, a roller or a trowel having a flat surface is employed. It is preferable to perform the pressing operation by the roller or the trowel, prior to applying the finishing coating, in order to obtain an excellent decorative layer.

The pressing operation can be performed by other similar methods without employing a roller or a trowel, although it is desirable to employ a roller such as that 5 shown in Figure 3.

The decorative layer, after being pressed by the roller or the trowel, is further cured and hardened. After the hardening, if necessary, the surface is treated with a synthetic resin enamel or lacquer which is coated one or several times on to the decorative layer.

The synthetic resin enamel is preferably a vinyl type resin paint, an acrylic type resin paint, an acrylic urethane type resin paint, but other synthetic resin enamels or lacquers can be used. The lustre of the decorative layer can be improved and the mechanical and chemical durability of the decorative layer can be improved by the finishing coating.

In accordance with the method of the invention, the epoxy resin emulsion is incorporated as a component of the decorative layer, whereby the diffusion velocity of the water is decreased and the evaporation of moisture from the decorative layer is therefore also decreased, and the formation of cracks in the decorative layer can be prevented by maintaining a desirable moisture content during the curing of the cement.

Moreover, the usual water sprinkling operation or intermediate treatment step at low

temperature can be eliminated because dehydration during the curing is minimized.

In the conventional reaction of the epoxy resin emulsion with the epoxy hardener, when water is present, the hardening is not completed, so that cross-linking rate is remarkably decreased and the adhesive strength is decreased.

However, in the mortar composition used in this invention, cement is mixed with the epoxy resin emulsion and the excess water of the epoxy resin emulsion is used as moisture required for curing the cement, so that the hardening of the epoxy resin can be completed, and the hardening velocity, the hardness and the adhesive strength at low temperature can be increased in comparison with a decorative layer prepared by using the epoxy resin emulsion above.

Since an epoxy resin emulsion is used, even though the base surface may be a water adsorbing concrete, e.g. a foam concrete, the fine particles of the epoxy resin emulsion permeate into the base surface to form thin layer slightly below the surface and further water permeation deep into the base is thereby prevented. Incidentally, when the base is a metal, it is advisable to treat the metal surface with a corrosion inhibitor primer.

The decorative layer prepared by using the conventional organic mortar is highly tacky in the semi-hardening condition, so that the pressing operation by the roller or the trowel is difficult. However, in accordance with this invention the tackiness can be decreased and the plasticity of the decorative layer is suitable after 0.5—6 hours and the convex parts of the decorative layer can be pressed without adhesion.

The following Examples will further illustrate the embodiments of this invention, and are not intended to be limiting in any manner.

Example 1.

The following mortar composition was kneaded thoroughly to a viscosity of 350 poise at 25°C, and was spray-coated through a

mortar gun having a nozzle diameter of 8 mm at a pressure of 5 kg/cm² on to a concrete surface to form a decorative layer having an uneven surface.

White cement	40 wt%	50
Aggregate (average 150 mesh)	22 wt%	
Water	15 wt%	
Pigment (TiO ₂)	3 wt%	
Tackifier (methylcellulose)	2 wt%	55
Epoxy resin (liquid epoxy resin having 2 terminal epoxy groups containing a non ionic surfactant)	6 wt%	
Epoxy hardener (diamine-dicarboxylic acid type polyamide)	6 wt%	60

After spray-coating the mortar composition, the decorative layer was cured at 20°C in an atmosphere having a relative humidity of 70% for one hour, and the projecting parts of the decorative layer were pressed with a roller having a smooth surface made of aluminum [shown in Figure 3] while the layer was still in the semi-hardened condition, so as to make the top of the projecting parts flat, and then the decorative layer was cured for 16 hours to fully harden it.

Three layers of an acryl-urethane type resin enamel were coated on the decorative layer as a finishing agent. The resulting decorative layer had a pattern similar to that as shown in Figure 2, and a special surface appearance having high gloss. A further decorative appearance can be imparted by providing a difference between the gloss of the flat top of projecting parts and the gloss of the non-pressed parts.

In the pressing operation by the roller, the mortar composition did not adhere to the roller and uniform pressing was thus easily obtained to provide uniform pressing. The following table, shows the results of tests of the characteristics of the decorative layer of this invention, and samples of conventional cement type decorative layers, solvent type epoxy resin decorative layers and thermoplastic resin type decorative layers.

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	Characteristics test	this invention	cement decorative layer	solvent epoxy resin layer	thermo-plastic resin layer
Adhesive strength	1] 5°C 80% RH 7 days	10-15 kg/cm ²	5 - 10 kg/cm ²	less than 5 kg/cm ²	less than 5 kg/cm ²
	2] weather meter 500 hours	17 - 19 ,,	7 - 10 kg/cm ²	peel from base	7 - 10 kg/cm ²
Gloss of decorative layer	1] convex	50 - 80%	less than 20	less than 20	less than 20
	2] concavo	2 - 6 ,,	less than 20	less than 20	less than 20
	3] difference of convex-concavo	48 - 78 ,,	0	0	0
Hardness	1] room temp.	54 - 61	50 - 60	45 - 70	less than 10
	2] 5°C 80% RH	46 - 51	40 - 50	5 - 46	2 - 7
Roller adhesive test	after coating the fluid composition after 1 hour	none	none	adhesive	adhesive
	2 hours	none	none	adhesive	adhesive
	3 hours	none	none	slight adhesive	slight adhesive
	4 hours	none	none	adhesive	adhesive

The sample of this invention to which the details given in the table above was prepared by the method of Example 1. The sample of the cement type decorative layer was prepared by spray-coating an aqueous emulsion containing 25% of vinylacetate resin on a concrete wall, and allowing it to dry for 1 hour and then spray-coating, by means of a mortar gun, a fluid composition prepared by kneading a mixture of 23.5 wt% of white Portland cement, 46.9 wt% of silica sand, 1.2 wt% of red pigment, 0.2 wt% of a hardening accelerator consisting mainly of calcium chloride, and 28.2 wt% of water. After 40 minutes a solution of about 50% of an acrylic ester in an alcohol was spray-coated on the layer and was then cured by leaving for 24 hours and then a finishing coating of a thinner solution of the acrylic ester was sprayed on the surface and was left for 1 week.

The solvent type epoxy resin layer was prepared by spray-coating a fluid composition containing 20 wt% of epoxy varnish, 5 wt% of coloring pigment, 40 wt% of powdery

aggregate powder, 15 wt% of asbestos powder and 20 wt% of epoxy hardener by a mortar gun, and leaving it for 1 week.

The thermoplastic resin layer was prepared by spray-coating a fluid composition containing 25 wt% of acryl type resin emulsion varnish, 55 wt% of aggregate powder, 5 wt% of pigment, 5 wt% of tackifier and the other additive, and 5 wt% of water, by a mortar gun and leaving it for 1 week.

In the characteristics tests, the adhesive strength was measured by Olsen tensile testing machine with a sample which was maintained at 5°C in an atmosphere having a relative humidity of 80% for 7 days, and the sample being maintained in a weather meter for 500 hours.

The hardness was measured by a Bar col hardness tester, wherein the sample was first maintained at ambient room temperature, and then at 5°C at a relative humidity of 80%. The gloss of the decorative layer was measured by the JIS [Japanese Industrial Standard] K 5400-6-7, 60° miller luster degree measurement.

The gloss of the projecting (convex) parts and the depressions (concavo parts) of each sample was measured at an angle of incidence of 60°C and at a receiving angle of 60°C 5 times for each sample by means of an electrophotometer, and the results were as calculated percentages [referring to the maximum and minimum values of the 5 measurements], when the gloss of the standard miller surface is 100.

The difference of the gloss at the convex parts and the concavo parts was shown as the difference of convex-concavo. The adhesive test was performed by coating a fluid composition of each sample and pressing it by rolling with an aluminum tubular roller having smooth surface (5 cm of diameter) which was washed, after 1, 2, 3 and 4 hours, and observing the adhesive condition of the fluid composition on the roller.

As stated above, in accordance with this invention, it is possible to provide the decorative layer having high adhesive strength without under-coating of the base, and having no formation of crack by sudden hardening, and it is not to require a water spray or a middle treatment for preventing dehydration on summer or at low temperature, and it is possible to provide the decorative layer having beautiful pattern and high water-resistance, alkali-resistance, and high adhesive strength without delay imparting suitable hardness and high adhesive strength, and accordingly, it is advantageous in industrial operation.

WHAT WE CLAIM IS:—

1. A method of preparing a decorative layer which comprises spray-coating a mortar composition containing 3—50 wt% of an epoxy resin and an epoxy hardener, 5—70 wt% of cement, 5—70 wt% of aggregate powder and 5—30 wt% of water onto the surface of a base to form a layer having an uneven surface, allowing the coating to achieve a semi-hardened condition and then pressing the surface of the layer to flatten the tops of the projecting parts.

2. A method as claimed in claim 1 which further comprises applying a finishing coating of a synthetic resin enamel or lacquer.

3. A method as claimed in any preceding claim, wherein the epoxy resin is introduced into the composition as an emulsion containing 40—60 wt% of the epoxy resin.

4. A method as claimed in any preceding claim wherein the ratio of the epoxy resin to the epoxy hardener in the composition is 1:0.5—1.5 by weight.

5. A method as claimed in any preceding claim, wherein the viscosity of the mortar composition is in the range of 100—500 poise at 25°C.

6. A method as claimed in any preceding claim wherein the spray-coated mortar is allowed to cure and harden for 0.5—6 hours to achieve the semi-hardened condition.

7. A method as claimed in any preceding claim wherein surface of the layer is pressed with a roller press.

8. A method as claimed in any preceding claim wherein the epoxy resin comprises a liquid epoxy resin.

9. A method as claimed in any preceding claim wherein the epoxy hardener comprises a polyamide.

10. A method as claimed in any preceding claim, wherein the cement is a hydraulic cement comprising white cement, Portland cement, alumina cement or mixtures thereof.

11. A method as claimed in any preceding claim wherein the aggregate powder consists of particles of 30 mesh (ASTM Standard) or smaller.

12. A method as claimed in any preceding claim, wherein the mortar composition is prepared by separately emulsifying the epoxy resin or a mixture of the epoxy resin and another type resin, and then admixing the epoxy hardener with the epoxy resin emulsion, the cement and the aggregate powder.

13. A method as claimed in any preceding claim, wherein the epoxy resin comprises a mixture of a liquid epoxy resin and a solid epoxy resin which is soluble to the liquid epoxy resin.

14. A method as claimed in any preceding claim, wherein the liquid epoxy resin has at least two terminal epoxy groups.

15. A method as claimed in claim 1 substantially as described herein with reference to the Examples.

16. A method as claimed in claim 1 substantially as described herein with reference to the accompanying drawings.

17. A decorative layer adhered to a base, prepared by a method as claimed in any preceding claim.

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The gloss of the projecting (convex) parts and the depressions (concavo parts) of each sample was measured at an angle of incidence of 60°C and at a receiving angle of 60°C 5 times for each sample by means of an electrophotometer, and the results were as calculated percentages [referring to the maximum and minimum values of the 5 measurements], when the gloss of the standard miller surface is 100.

The difference of the gloss at the convex parts and the concavo parts was shown as the difference of convex-concavo. The adhesive test was performed by coating a fluid composition of each sample and pressing it by rolling with an aluminum tubular roller having smooth surface (5 cm of diameter) which was washed, after 1, 2, 3 and 4 hours, and observing the adhesive condition of the fluid composition on the roller.

As stated above, in accordance with this invention, it is possible to provide the decorative layer having high adhesive strength without under-coating of the base, and having no formation of crack by sudden hardening, and it is not to require a water spray or a middle treatment for preventing dehydration on summer or at low temperature, and it is possible to provide the decorative layer having beautiful pattern and high water-resistance, alkali-resistance, and high adhesive strength without delay imparting suitable hardness and high adhesive strength, and accordingly, it is advantageous in industrial operation.

WHAT WE CLAIM IS:—

1. A method of preparing a decorative layer which comprises spray-coating a mortar composition containing 3—50 wt% of an epoxy resin and an epoxy hardener, 5—70 wt% of cement, 5—70 wt% of aggregate powder and 5—30 wt% of water onto the surface of a base to form a layer having an uneven surface, allowing the coating to achieve a semi-hardened condition and then pressing the surface of the layer to flatten the tops of the projecting parts.

2. A method as claimed in claim 1 which further comprises applying a finishing coating of a synthetic resin enamel or lacquer.

3. A method as claimed in any preceding claim, wherein the epoxy resin is introduced into the composition as an emulsion containing 40—60 wt% of the epoxy resin.

4. A method as claimed in any preceding claim wherein the ratio of the epoxy resin to the epoxy hardener in the composition is 1:0.5—1.5 by weight.

5. A method as claimed in any preceding claim, wherein the viscosity of the mortar composition is in the range of 100—500 poise at 25°C.

6. A method as claimed in any preceding claim wherein the spray-coated mortar is allowed to cure and harden for 0.5—6 hours to achieve the semi-hardened condition.

7. A method as claimed in any preceding claim wherein surface of the layer is pressed with a roller press.

8. A method as claimed in any preceding claim wherein the epoxy resin comprises a liquid epoxy resin.

9. A method as claimed in any preceding claim wherein the epoxy hardener comprises a polyamide.

10. A method as claimed in any preceding claim, wherein the cement is a hydraulic cement comprising white cement, Portland cement, alumina cement or mixtures thereof.

11. A method as claimed in any preceding claim wherein the aggregate powder consists of particles of 30 mesh (ASTM Standard) or smaller.

12. A method as claimed in any preceding claim, wherein the mortar composition is prepared by separately emulsifying the epoxy resin or a mixture of the epoxy resin and another type resin, and then admixing the epoxy hardener with the epoxy resin emulsion, the cement and the aggregate powder.

13. A method as claimed in any preceding claim, wherein the epoxy resin comprises a mixture of a liquid epoxy resin and a solid epoxy resin which is soluble to the liquid epoxy resin.

14. A method as claimed in any preceding claim, wherein the liquid epoxy resin has at least two terminal epoxy groups.

15. A method as claimed in claim 1 substantially as described herein with reference to the Examples.

16. A method as claimed in claim 1 substantially as described herein with reference to the accompanying drawings.

17. A decorative layer adhered to a base, prepared by a method as claimed in any preceding claim.

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