

PATENT SPECIFICATION

(11) 1 518 168

- (21) Application No. 47856/75 (22) Filed 20 Nov. 1975
 (23) Complete Specification filed 15 Nov. 1976
 (44) Complete Specification published 19 July 1978
 (51) INT CL² C08G 59/56
 (52) Index at acceptance
 C3B 1D2C 1N1X 1N6D1 1N6D2 1N6D4 1N6D5 1N6D8 E1
 EX
 (72) Inventor RICHARD BRIAN TILLYER



(54) EPOXY COMPOSITIONS

(71) We, CHEMICAL BUILDING PRODUCTS LIMITED, of Cleveland Road, Hemel Hempstead, Hertfordshire, England, an English Company, 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 hardeners for epoxy resin compositions, especially, but not exclusively, for use as industrial flooring screeds.

A problem with industrial epoxy resin flooring compositions is the odour evolved. Gradually an epoxy resin flooring screed will be laid when the building is empty, for example at a weekend or public holiday, with the hope that the odour will have diminished to an acceptable level by the time the building is in use again. For certain applications, however, the odour, no matter how slight, cannot be tolerated since the product being made in the building may be tainted by the odour, particularly where foodstuffs are being produced.

In the case of flooring compositions based on epoxy resin, the odour is generally caused by the amine hardener composition used to harden the resin. This invention provides a hardener composition, the use of which will reduce or avoid the odour and tainting problem.

Another problem experienced with industrial epoxy resin and other flooring compositions arises from the need to steam clean floors in areas where cleanliness is especially important, such in food-preparation and pharmaceuticals factories. Repeated steam cleaning can give rise to separation of the composition from the underlying, usually concrete, substrate, and hence breaking up the surface.

The hardener composition must also be of sufficiently low viscosity to provide good trowelling properties and wetting during application.

The use of aliphatic polyamines alone to cure epoxy resins is well known, but such compounds give rise to an unacceptable odour, in use.

Accordingly, the invention provides a liquid hardener composition for use in curing an epoxy resin, comprising a blend of a cycloaliphatic amine and a low odour product formed by the reaction between a polyether urethane having free hydroxyl groups and an aliphatic polyamine curing agent for the epoxy resin.

For ease of workability of the resin and hardener mix, the viscosity of the hardener composition should preferably be about 1 to about 25 poises at 25°C, about 5 to about 15 poises being especially suitable.

In this hardener composition, the polyether urethane is pre-reacted with the aliphatic polyamine to form a product which is of low odour and which imparts flexibility to the cured epoxy resin. The cycloaliphatic amine is added to ensure a balance between toughness and flexibility of the cured epoxy resin.

Preferably the aliphatic amine, the polyether urethane and the cycloaliphatic amine are used in the proportion 5:5:2 by weight, but other proportions, such as 2:2:1 are usable.

The aliphatic amine is preferably a substituted aliphatic diamine. Preferred aliphatic polyamines are those having three reactive hydrogen atoms per molecule and a hydrogen active equivalent value of 30 to 60, most preferably 50, where the hydrogen active equivalent value is defined as the number of grammes of the amine required to react with the epoxy equivalent weight (E.E.W.) in grammes of epoxy resin. The aliphatic polyamine will usually be a liquid, preferably having a viscosity of about 5 to 6 poises at 25°C for example, N-benzylethylene diamine being especially suitable.

The cycloaliphatic amine is preferably a liquid having a viscosity of about 148 poises at 20°C.

The polyether urethane is preferably a solvent-free high viscosity (approximately 1200 poises at 20°C) liquid, which may be thinned using suitable odour-free or low-odour solvents.

The invention also provides a method of making a liquid hardener composition for use in curing an epoxy resin, which comprises blending together a polyether urethane having free hydroxyl groups and an aliphatic polyamine, allowing the blend to react to form a low-odour product, and mixing a cycloaliphatic amine with the product.

Preferably, the blend of the polyether urethane and the aliphatic polyamine is allowed to stand for at least 2 hours to react to form the low-odour product before addition of the cycloaliphatic amine. Equal proportions by weight of the polyether urethane and the aliphatic polyamine may be blended, and the product is mixed with the cycloaliphatic amine in the weight ratio of 4 to 5:1.

The invention includes a self-setting composition comprising an epoxy resin composition in admixture with a liquid hardener composition according to the invention. The resin is suitably one having an epoxide equivalent weight (E.E.W.) of 200.

A plasticiser will usually be present in the resin composition; examples are dibutyl phthalate and benzyl octyl adipate. A silane resin may also be added, to improve bonding between the self-setting composition and a substrate upon which it is being laid, particularly where the composition is being used as a flooring screed. Pigments and other known additives, such as anti-settle agents, may also be added.

Preferably the epoxy resin composition and the liquid hardener are used in a proportion of from 1 to 10:1 by weight, a proportion of 3:1 being especially suitable.

For flooring purposes, it is usual to add to the self-setting composition suitable aggregates, and this may be done in a ratio of from 3 to 15 parts by weight, preferably of from 6 to 7 parts by weight, per part of the composition.

Best results in flooring applications are achieved by the use of a priming coat of a self-setting composition in accordance with the invention, in which the hardener composition is diluted, before addition to the epoxy resin composition, with a low odour solvent. Preferably the solvent is added to the hardener composition in a weight ratio of 1:6. The priming coat (which will normally be unpigmented and exclude aggregate), may be applied to a concrete substrate for example, and allowed to dry before laying of the self-setting composition of the invention. A sealer coat may be applied to the surface of the floor to provide a final working floor surface. The sealer coat is substantially the same as the priming coat, but may include a pigment, and a small amount of aggregate to improve the non-slip character of the floor.

The invention is illustrated by the following Examples, in which all parts are by weight.

EXAMPLE 1

A liquid epoxy resin composition was made up from the following:—

	parts
epoxy resin (EEW of 200)	16
dibutyl phthalate (plasticiser)	3
pigment (red iron oxide)	1

A liquid hardener composition was made up by blending the following and leaving the blend to stand for 24 hours:—

	parts
N-benzylethylene diamine (aliphatic polyamine)	2
polyether urethane (Desmocap 11)	2
("Desmocap 11" is a trade mark of Bayer A.G.)	

The product was blended with 1 part of the cycloaliphatic amine HY 1537 ("HY 1537" is a product of C.I.B.A. Ltd.).

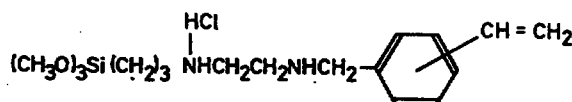
The resin composition and hardener composition were blended by machine in a weight ratio of 3:1 and then mixed with a screeding aggregate in a weight ratio of 1:6. The formed self-setting composition was applied to an industrial floor in a food factory to a thickness of 6 mm and left for 24 hours to harden sufficiently to carry traffic. Food samples were left exposed on tables in the factory during application of the composition and were subsequently found to be taint-free and no significant level of amine odour was detectable at any stage.

EXAMPLE 2

A liquid composition was made up by blending the following:—

		parts	
5	Epoxy resin (EEW of 200)	100	
	dibutyl phthalate (plasticiser)	7	5
	silane resin solution	4	
	Red Iron Oxide powder pigment	5	
	Anti-settle agent	0.7	

The silane resin solution used was a 40% solution in methanol of a styryl amine functional trimethoxysilane having the formula:—



A hardener composition was then prepared by blending 22 parts of N-butyl-ethylene diamine with 22 parts of polyether urethane (Desmocap 11) and allowing the blend to stand for 2 hours. 8.8 parts of cycloaliphatic amine (HY 1537) were then blended in.

The resin composition and hardener composition were blended by machine in a weight ratio of 2:1 and then mixed with a screeding aggregate consisting of fine silica sands having the following size composition:—

20	40% passing B.S. 14 mesh	20
	40% passing B.S. 25 mesh	
	20% passing B.S. 60 mesh	

in the weight ratio of 6.35 parts of aggregate to one part of the blend, to form a screeding composition.

A concrete floor substrate had previously been coated with a priming composition of a self-setting composition made up in the same way as for the screeding composition, but excluding the pigment and the aggregate and with 8.8 parts of a low-odour solvent, Shellsol T, added to the hardener composition before its addition to the resin composition. The priming coat was allowed to dry before application of the screed.

The screeding composition was then applied to the substrate in a layer 5—6mm thick and allowed to cure for 12 hours. A sealer composition was then prepared in the same manner as the priming coat, but with the addition to the resin composition of 5 parts by weight of red iron oxide powder pigment. A minor amount of aggregate was added to the mixed sealer composition to improve the non-slip character of the surface. The sealer composition was then applied as a thin coat to the cured screeding composition and allowed to cure.

Very little odour was detected during or after laying of the floor. The floor was subsequently steam cleaned in known manner, but showed no sign of deterioration or detachment from the substrate.

WHAT WE CLAIM IS:—

1. A liquid hardener composition for use in curing an epoxy resin, comprising a blend of a cycloaliphatic amine and a low-odour product formed by the reaction between a polyether urethane having free hydroxyl groups and an aliphatic polyamine curing agent for the epoxy resin.

2. A hardener composition according to claim 1, having a viscosity of 1 to 25 poises at 25°C.

3. A hardener composition according to claim 2, having a viscosity of 5 to 15 poises at 25°C.

4. A hardener composition according to claim 1, 2, or 3, which is formed from, by weight, 5 parts of the aliphatic polyamine, 5 parts of the polyether urethane, and 2 parts of the cycloaliphatic amine.

5. A hardener composition according to any preceding claim, in which the aliphatic polyamine is a substituted aliphatic diamine.

6. A hardener composition according to any preceding claim, in which the aliphatic polyamine has three reactive hydrogen atoms per molecule and a hydrogen active equivalent value as hereinbefore defined of 30 to 60.



(11)

3

1,518,168

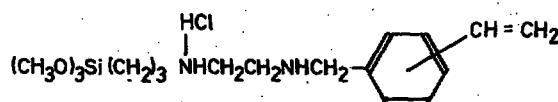
3

EXAMPLE 2

A liquid composition was made up by blending the following:—

		parts	
5	Epoxy resin (BEW of 200)	100	
	dibutyl phthalate (plasticiser)	7	5
	silane resin solution	4	
	Red Iron Oxide powder pigment	5	
	Anti-settle agent	0.7	

10 The silane resin solution used was a 40% solution in methanol of a stryryl amine functional trimethoxysilane having the formula:— 10



15 A hardener composition was then prepared by blending 22 parts of N-butyl-ethylene diamine with 22 parts of polyether urethane (Desmocap 11) and allowing the blend to stand for 2 hours. 8.8 parts of cycloaliphatic amine (HY 1537) were then blended in. 15

The resin composition and hardener composition were blended by machine in a weight ratio of 2:1 and then mixed with a screeding aggregate consisting of fine silica sands having the following size composition:—

20	40% passing B.S. 14 mesh	20
	40% passing B.S. 25 mesh	
	20% passing B.S. 60 mesh	

in the weight ratio of 6.35 parts of aggregate to one part of the blend, to form a screeding composition.

25 A concrete floor substrate had previously been coated with a priming composition of a self-setting composition made up in the same way as for the screeding composition, but excluding the pigment and the aggregate and with 8.8 parts of a low-odour solvent, Shellsol T, added to the hardener composition before its addition to the resin composition. The priming coat was allowed to dry before application of the screed. 25

30 The screeding composition was then applied to the substrate in a layer 5–6mm thick and allowed to cure for 12 hours. A sealer composition was then prepared in the same manner as the priming coat, but with the addition to the resin composition of 5 parts by weight of red iron oxide powder pigment. A minor amount of aggregate was added to the mixed sealer composition to improve the non-slip character of the surface. The sealer composition was then applied as a thin coat to the cured screeding composition and allowed to cure. 30

35 Very little odour was detected during or after laying of the floor. The floor was subsequently steam cleaned in known manner, but showed no sign of deterioration or detachment from the substrate. 35

WHAT WE CLAIM IS:—

40 1. A liquid hardener composition for use in curing an epoxy resin, comprising a blend of a cycloaliphatic amine and a low-odour product formed by the reaction between a polyether urethane having free hydroxyl groups and an aliphatic polyamine curing agent for the epoxy resin. 40

45 2. A hardener composition according to claim 1, having a viscosity of 1 to 25 poises at 25°C. 45

3. A hardener composition according to claim 2, having a viscosity of 5 to 15 poises at 25°C.

50 4. A hardener composition according to claim 1, 2, or 3, which is formed from, by weight, 5 parts of the aliphatic polyamine, 5 parts of the polyether urethane, and 2 parts of the cycloaliphatic amine. 50

5. A hardener composition according to any preceding claim, in which the aliphatic polyamine is a substituted aliphatic diamine.

55 6. A hardener composition according to any preceding claim, in which the aliphatic polyamine has three reactive hydrogen atoms per molecule and a hydrogen active equivalent value as hereinbefore defined of 30 to 60. 55

12

1,518,168

4

7. A hardener composition according to claim 6, in which the hydrogen active equivalent value of the aliphatic polyamine is 50.

8. A hardener composition according to any preceding claim, in which the aliphatic polyamine is a liquid having a viscosity of 5 to 6 poises at 25°C.

9. A hardener composition according to any preceding claim, in which the aliphatic polyamine is N-benzylethylene diamine.

10. A hardener composition according to any preceding claim, in which the cycloaliphatic amine is a liquid having a viscosity of about 140 poises at 20°C.

11. A liquid hardener composition according to claim 1 for use in curing an epoxy resin substantially as described in Example 1 or 2.

12. A method of making a liquid hardener composition for use in curing an epoxy resin, which comprises blending together a polyether urethane having free hydroxyl groups and an aliphatic polyamine, allowing the blend to react to form a low odour product and mixing a cycloaliphatic amine with the product.

13. A method according to claim 12, in which the blend of the polyether urethane and the aliphatic polyamine is allowed to stand for at least 2 hours before addition of the cycloaliphatic amine.

14. A method according to claim 12 or 13, in which equal proportions by weight of the polyether urethane and the aliphatic polyamine are blended, and the resultant product is mixed with the cycloaliphatic amine in the weight ratio of 4 to 5:1.

15. A method of making a liquid hardener composition according to claim 12 substantially as described in Example 1 or 2.

16. A liquid hardener composition for use in curing an epoxy resin, when made by a method according to any of claims 11 to 14.

17. A self-setting composition comprising an epoxy resin composition in admixture with a liquid hardener composition according to any of claims 1 to 11, or 16.

18. A composition according to claim 17, in which the epoxy resin has an epoxide equivalent weight of 200.

19. A composition according to claim 17 or 18, in which the resin composition includes a plasticiser.

20. A composition according to claim 19, in which the plasticiser is dibutyl phthalate.

21. A composition according to claim 19, in which the plasticiser is benzyl octyl adipate.

22. A composition according to any of claims 17 to 21, in which the resin composition includes a silane resin.

23. A composition according to any of claims 17 to 22, which comprises, by weight, 1 to 10 parts of the resin composition and 1 part of the hardener composition.

24. A composition according to claim 23, which comprises, by weight, 3 parts of the resin composition and 1 part of the hardener composition.

25. A composition according to any of claims 17 to 24, which comprises a screeding aggregate.

26. A composition according to claim 25, which comprises, by weight, 3 to 15 parts of aggregate and 1 part of the admixture of the resin composition and hardener composition.

27. A composition according to claim 26, which comprises, by weight, 6 to 7 parts of aggregate and 1 part of the admixture of the resin composition and hardener composition.

28. A self-setting composition according to claim 17, substantially as described in Example 1 or 2.

29. A floor, when surfaced with a self-setting composition according to any of claims 17 to 28.

LAURENCE SHAW,
Registered Patent Agent,
Agent for the Applicants.

Printed for Her Majesty's Stationery Office, by the Courier Press, Leamington Spa, 1978
Published by The Patent Office, 25 Southampton Buildings, London, WC2A 1AY, from
which copies may be obtained.