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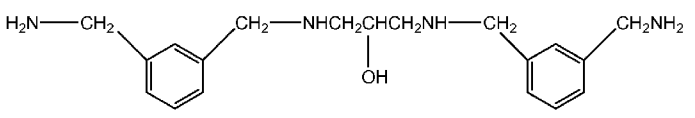
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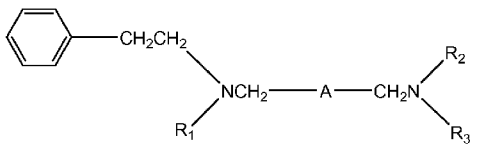
— *with international search report (Art. 21(3))*

— *before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments (Rule 48.2(h))*

(54) **Title**: AMINE CURABLE EPOXY RESIN COMPOSITION



(I)



(II)

(57) **Abstract**: The present invention relates to an amine curable epoxy resin composition comprising:(A) epoxy resin component comprising:8-31 wt% of diglycidyl ether of bisphenol A,3-14 wt% of diglycidyl ether of bisphenol F,1-5 wt% of monoglycidylether,0.6-5 wt% of alkylsulphonic phenyl ester,0.16-1 wt% of wetting and dispersing agent,0.16-1 wt% of defoamer, and5-40 wt% of barium sulfate;(B) hardener component comprising:3-18 wt% of first amine based composition, and 2-12 wt% of second amine based composition, wherein the first amine based composition comprises N,N'-bis(3-aminomethylbenzyl)-2-hydroxy-trimethylenediamine represented by the following formula (I) and m-xylylenediamine, and the second amine based composition comprises the reaction products of m-xylylenediamineand styrene represented by the following formula (II) and m-xylylenediamine, wherein A is phenylene, and each of R_1, $R_2$and R_3 independently represents hydrogen or phenylethyl; wherein the sum of each component content is 100wt%, and each weight percentage is based on the total weight of the amine curable epoxy resin composition. The invention also relates to use of the amine curable epoxy resin compositionas primer, topcoat and screeds for application on concrete.

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Amine curable epoxy resin composition

Technical field

The invention relates to an amine curable epoxy resin composition. The invention also relates to use of the amine curable epoxy resin composition as primer, topcoat and screeds for application on concrete.

Background of art

Amine curable epoxy resin compositions are used broadly in the floorings and coatings. For the moment, amine curable epoxy resin compositions in the market have longer Return to Service time or shorter pot life at room temperature, which causes them not able to being used easily at room temperature. GB 2 286 592 A disclosed an amine curable epoxy resin composition, wherein the composition with hardener GASMAMINE 328 has compressive strength of 30.5 MPa after application for 24 hours. However, there is still a need that amine curable epoxy resin compositions having higher compressive strength, shorter Return to Service time and longer pot life are provided.

Invention summary

Accordingly, the invention provides an amine curable epoxy resin composition comprising:

(A) epoxy resin component comprising:

8-31 wt% of diglycidyl ether of bisphenol A,

3-14 wt% of diglycidyl ether of bisphenol F,

1-5 wt% of monoglycidylether,

0.6-5 wt% of alkylsulphonic phenyl ester,

0.16-1 wt% of wetting and dispersing agent,

0.16-1 wt% of defoamer, and

5-40 wt% of barium sulfate;

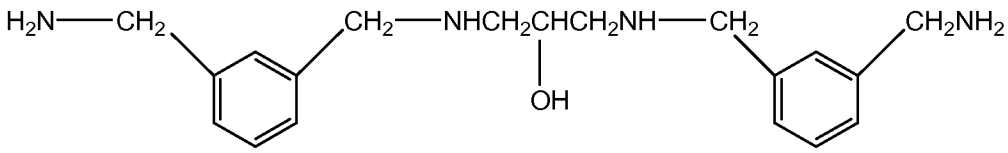
(B) hardener component comprising:

3-18 wt% of first amine based composition, and

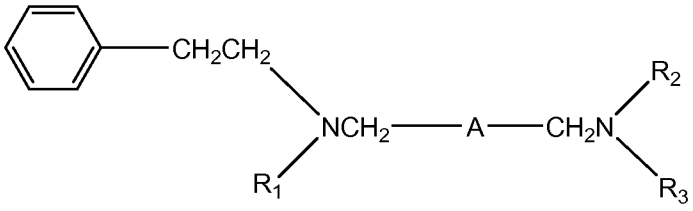
2-12 wt% of second amine based composition,

wherein the first amine based composition comprises N,N'-bis(3-aminomethylbenzyl)-2

-hydroxytrimethylenediamine represented by the following formula (I) and m-xylylenediamine, and the second amine based composition comprises the reaction products of m-xylylenediamine and styrene represented by the following formula (II) and m-xylylenediamine,



(I)

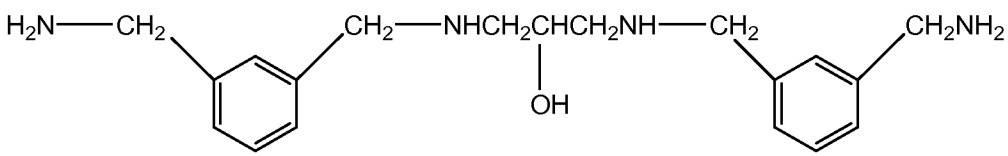


(II)

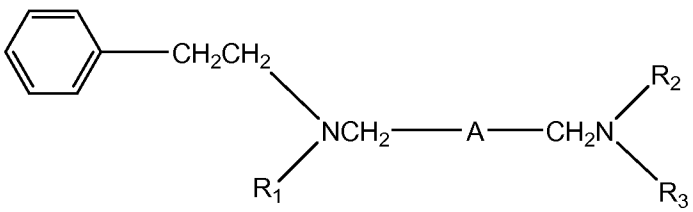
wherein A is phenylene, and each of R_1, R_2 and R_3 independently represents hydrogen or phenylethyl;

wherein the sum of each component content is 100wt%, and each weight percentage is based on the total weight of the amine curable epoxy resin composition.

The invention also provides a hardener comprising 20-90 wt% of first amine based composition and 10-80 wt% of second amine based composition, wherein the first amine based composition comprises N,N'-bis(3-aminomethylbenzyl)-2-hydroxytrimethylenediamine represented by the following formula (I) and m-xylylenediamine, and the second amine based composition comprises the reaction products of m-xylylenediamine and styrene represented by the following formula (II) and m-xylylenediamine,



(I)



(II)

wherein A is phenylene, and each of R_1, R_2 and R_3 independently represents hydrogen or phenylethyl;

wherein the sum of each component content is 100wt%, and each weight percentage is based on the total weight of the first amine based composition and the second amine based composition.

The invention also provides use of the amine curable epoxy resin composition as primer, topcoat and screeds for application on concrete.

The amine curable epoxy resin composition of the present invention has pot life of more than 25 minutes, Return to Service time of less than 12 hours and compressive strength of more than 80 MPa after application for 20 hours at room temperature.

Embodiments

In one embodiment of the present invention, the invention provides an amine curable epoxy resin composition comprising:

(A) epoxy resin component comprising:

8-31 wt% of diglycidyl ether of bisphenol A,

3-14 wt% of diglycidyl ether of bisphenol F,

1-5 wt% of monoglycidylether,

0.6-5 wt% of alkylsulphonic phenyl ester,

0.16-1 wt% of wetting and dispersing agent,

0.16-1 wt% of defoamer, and

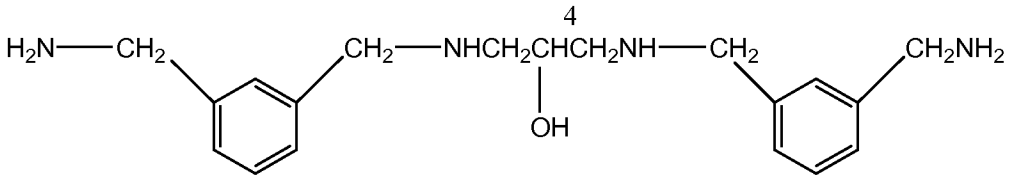
5-40 wt% of barium sulfate;

(B) hardener component comprising:

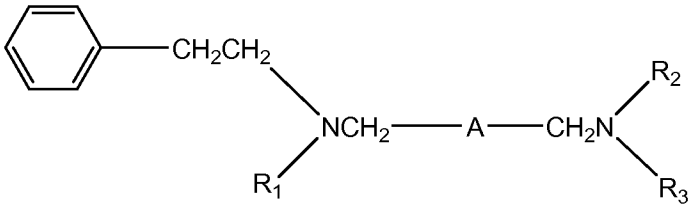
3-18 wt% of first amine based composition, and

2-12 wt% of second amine based composition,

wherein the first amine based composition comprises N,N'-bis(3-aminomethylbenzyl)-2-hydroxytrimethylenediamine represented by the following formula (I) and m-xylylenediamine, and the second amine based composition comprises the reaction products of m-xylylenediamine and styrene represented by the following formula (II) and m-xylylenediamine,



(I)

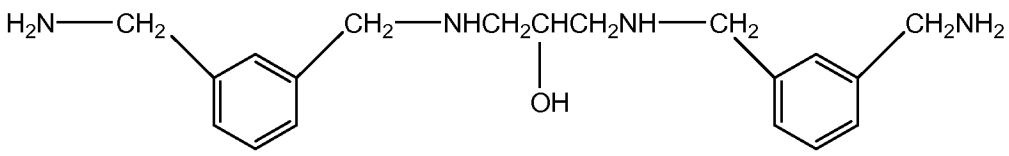


(II)

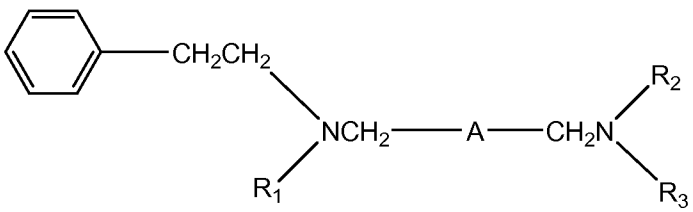
wherein A is phenylene, and each of R_1, R_2 and R_3 independently represents hydrogen or phenylethyl;

wherein the sum of each component content is 100wt%, and each weight percentage is based on the total weight of the amine curable epoxy resin composition.

In one embodiment of the present invention, the invention provides a hardener comprising 20-90 wt% of first amine based composition and 10-80 wt% of second amine based composition, wherein the first amine based composition comprises N,N'-bis(3-aminomethylbenzyl)-2 -hydroxytrimethylenediamine represented by the following formula (I) and m-xylylenediamine, and the second amine based composition comprises the reaction products of m-xylylenediamine and styrene represented by the following formula (II) and m-xylylenediamine,



(I)



(II)

wherein A is phenylene, and each of R_1, R_2 and R_3 independently represents hydrogen or phenylethyl;

wherein the sum of each component content is 100wt%, and each weight percentage is based on the total weight of the first amine based composition and the second amine based composition.

In one embodiment of the present invention, N,N'-bis(3-aminomethylbenzyl)-2-hydroxytrimethylenediamine represented by the formula (I) is a reaction product of m-xylylenediamine and epichlorhydrin, which can be prepared by known process in the art.

In one embodiment of the present invention, the reaction products of m-xylylenediamine and styrene represented by the formula (II) are prepared according to the process described in CN03149428.5, for example see synthesis example 1.

In one embodiment of the present invention, the amine curable epoxy resin composition may further comprise silica sand. In preferred embodiment of the present invention, the particle size of silica sand is 200 meshes.

In preferred embodiment of the present invention, the content of N,N'-bis(3-aminomethylbenzyl)-2-hydroxytrimethylenediamine is 72-76wt% of the first amine based composition and the content of m-xylylenediamine is 24-28wt% of the first amine based composition in the first amine based composition. More preferably, the content of N,N'-bis(3-aminomethylbenzyl)-2-hydroxytrimethylenediamine is about 75wt% of the first amine based composition and the content of m-xylylenediamine is about 25wt% of the first amine based composition in the first amine based composition. Said first amine based composition is for example GASKAMINE 328 commercially available from Mitsubishi Gas Chemical Company, Inc.

In preferred embodiment of the present invention, the content of m-xylylenediamine is less than 1wt% of the second amine based composition in the second amine based composition. Said second amine based composition is for example GASKAMINE 240 commercially available from Mitsubishi Gas Chemical America, Inc.

In another embodiment of the present invention, said hardener component may further comprise CeTePox EW99 H which is a formulation based on alkyl polyamines, and CeTePox EW99 H is for example commercially available from CTP Chemicals and Technologies for Polymers GmbH.

In one embodiment of the present invention, the hardener component may further comprise benzyl alcohol.

In one embodiment of the present invention, the diglycidyl ether of bisphenol A is for example GELR 128 commercially available from Epoxy Base Electronic Material Corporation Limited. The

diglycidyl ether of bisphenol F is for example Epikote 862 commercially available from Momentive Specialty Chemicals Inc..

In one embodiment of the present invention, monoglycidylether is for example Araldite DY-E commercially available from Huntsman.

In one embodiment of the present invention, the alkyl in the alkylsulphonic phenyl ester is C12 to C18. More preferably, the alkylsulphonic phenyl ester is dodecylsulphonic phenyl ester. The alkylsulphonic phenyl ester is for example MESAMOIL commercially available from LANXESS.

In preferred embodiment of the present invention, the wetting and dispersing agent is a solution of copolymer with acidic groups, and is for example DISPERBYK-110 commercially available from BYK Additives & Instrument.

In preferred embodiment of the present invention, the defoamer is a defoaming polymer mixture without organic silicon, and is for example BYK 1790 commercially available from BYK Additives & Instrument.

In preferred embodiment of the present invention, the particle size of barium sulfate is preferably 325 meshes.

In one embodiment of the present invention, the amine curable epoxy resin composition can be prepared as follows: (A) epoxy resin component is added into mixer at room temperature, such as high speed mixing machine, and mixed until homogeneity. Then, (B) hardener component is added into the above mixture and mixed until homogeneity. Optionally, silica sand can be added into the mixture of (A) epoxy resin component and (B) hardener component and mixed until homogeneity. Said high speed mixing machine is known to those skilled in the art.

In one embodiment of the present invention, (A) epoxy resin component and (B) hardener component can be packaged separately, and (A) epoxy resin component and (B) hardener component and optionally silica sand are mixed homogeneously before application.

The invention also relates to use of the amine curable epoxy resin composition as primer, topcoat and screeds for application on concrete. The amine curable epoxy resin composition can be applied by processes known to those skilled in the art, for example by using notched trowel, roller and spraying machine etc.

“Return to Service time” used herein represents a time period during which its hardness

achieves Shore D 70 after the amine curable epoxy resin composition is applied on concrete with moisture content of 5wt% at room temperature.

"Pot life" used herein is measured according to method 1 of ISO 10364.

"Compressive strength" used herein is measured according to ASTM C 109.

All percentages are mentioned by weight unless otherwise indicated.

Examples

The present invention is now further illustrated by reference to the following examples, however, the examples are used for the purpose of explanation and not intended to limit the scopes of the invention.

Example 1

125g of diglycidyl ether of bisphenol A (GELR 128), 54g of diglycidyl ether of bisphenol F (Epikote 862), 15g of monoglycidylether (Araldite DY-E), 12g of alkylsulphonic phenyl ester (MESAMOIL), 3g of wetting and dispersing agent (DISPERBYK-110), 3g of defoamer (BYK 1790) and 138g of barium sulfate are added into high speed mixing machine and mixed until homogeneity , and mixture 1 is obtained. Then, the mixture of 40.2g of GASKAMINE 328 and 27.2g of GASKAMINE 240 is added into the mixture 1 and mixed until homogeneity, and mixture 2 is obtained.

Mixture 2 is applied by rolling as primer on concrete with moisture content of 5wt% at the temperature of 23±2°C and relative humidity of 50% , and wet film thickness (WFT) is 200 μm.

Mixture 2 and film prepared thereof are measured. The results are listed in table 1.

Table 1

Property	Viscosity of mixture (cps)	Pot life (minute)	Return to Service time (hour)
Example 1	1517	43	7

835g of 200 meshes silica sand is added into the mixture 2 and mixed until homogeneity. Then, the resulted mixture is applied as screeds on concrete with moisture content of 5wt% at the temperature of 23±2°C and relative humidity of 50%, and compressive strength is measured as shown in table 2.

Table 2

curing time (hour)	compressive strength (MPa)
20	89.1
24	88.6
168	95.6

Example 2

Mixture of 40.2g of GASKAMINE 328, 27.7g of GASKAMINE 240 and 10g of benzyl alcohol is added into the mixture 1 according to example 1 and mixed until homogeneity, and mixture 3 is obtained.

Mixture 3 is applied by rolling as primer on concrete with moisture content of 5wt% at the temperature of 23±2°C and relative humidity of 50% , and wet film thickness (WFT) is 0.5 millimeter.

Mixture 3 and film prepared thereof are measured. The results are listed in table 3.

Table 3

Property	Viscosity of mixture (cps)	Pot life (minute)	Return to Service time (hour)
Example 2	1037	36	8

855g of 200 meshes silica sand is added into the mixture 3 and mixed until homogeneity. Then, the resulted mixture is applied as screeds on concrete with moisture content of 5wt% at the temperature of 23±2°C and relative humidity of 50%, and compressive strength is measured as shown in table 4.

Table 4

curing time (hour)	compressive strength (MPa)
20	98.5
24	98.5
168	115.8

Example 3

Mixture of 40.2g of GASKAMINE 328, 13.4g of GASKAMINE 240, 13.4g of CeTePox EW99 H and 10g of benzyl alcohol is added into the mixture 1 according to example 1 and mixed until homogeneity, and mixture 4 is obtained.

Mixture 4 is applied by scraping as topcoat on concrete with moisture content of 5wt% at the temperature of 23±2°C and relative humidity of 50% , and wet film thickness (WFT) is 2.0 millimeters.

Mixture 4 and film prepared thereof are measured. The results are listed in table 5 and table 6.

Table 5

Property	Viscosity of mixture (cps)	Pot life (minute)	Return to Service time (hour)
Example 3	1037	27	8

Table 6

curing time (hour)	compressive strength (MPa)
20	140
24	140
168	140

Example 4

854g of 200 meshes silica sand is added into the mixture 4 according to example 3 and mixed until homogeneity. Then, the resulted mixture is applied as screeds on concrete with moisture content of 5wt% at the temperature of 23±2°C and relative humidity of 50%, and compressive strength is measured as shown in table 7.

Table 7

curing time (hour)	compressive strength (MPa)
20	91.1
24	93.5
168	107.5

Example 5

Mixture of 40.2g of GASKAMINE 328, 27.7g of GASKAMINE 240 and 20g of benzyl alcohol is added into the mixture 1 according to example 1 and mixed until homogeneity, and mixture 5 is obtained.

Mixture 5 is applied by scraping as topcoat on concrete with moisture content of 5wt% at the temperature of 23±2°C and relative humidity of 50% , and wet film thickness (WFT) is 3.0 millimeters.

Mixture 5 and film prepared thereof are measured. The results are listed in table 8.

Table 8

Property	Viscosity of mixture (cps)	Pot life (minute)	Return to Service time (hour)
Example 5	870	33	12

874g of 200 meshes silica sand is added into the mixture 5 and mixed until homogeneity. Then, the resulted mixture is applied as screeds on concrete with moisture content of 5wt% at the

temperature of 23±2°C and relative humidity of 50%, and compressive strength is measured as shown in table 9.

Table 9

curing time (hour)	compressive strength (MPa)
20	80.3
24	81.9
168	98.2

It will be apparent to those skilled in the art that various modifications and variations can be made in the present invention without departing from the scope or spirit of the invention. Thus, it is intended that the present invention cover such modifications and variations as come within the scope of the appended claims and their equivalents.

Claims

1. An amine curable epoxy resin composition comprising:

(A) epoxy resin component comprising:

8-31 wt% of diglycidyl ether of bisphenol A,

3-14 wt% of diglycidyl ether of bisphenol F,

1-5 wt% of monoglycidylether,

0.6-5 wt% of alkylsulphonic phenyl ester,

0.16-1 wt% of wetting and dispersing agent,

0.16-1 wt% of defoamer, and

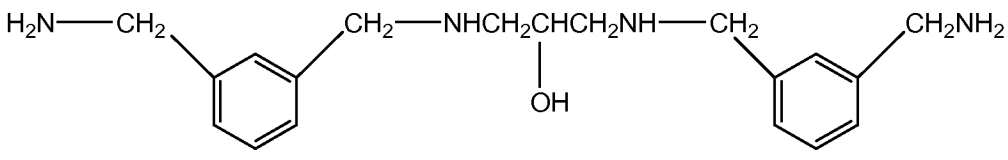
5-40 wt% of barium sulfate;

(B) hardener component comprising:

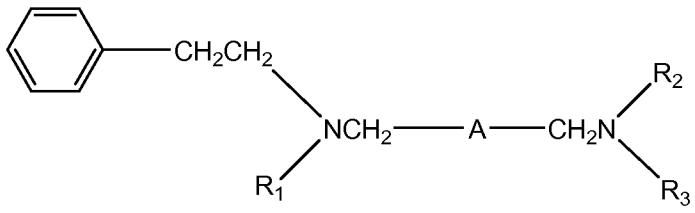
3-18 wt% of first amine based composition, and

2-12 wt% of second amine based composition,

wherein the first amine based composition comprises N,N'-bis(3-aminomethylbenzyl)-2-hydroxytrimethylenediamine represented by the following formula (I) and m-xylylenediamine, and the second amine based composition comprises the reaction products of m-xylylenediamine and styrene represented by the following formula (II) and m-xylylenediamine,



(I)



(II)

wherein A is phenylene, and each of R_1, R_2 and R_3 independently represents hydrogen or phenylethyl;

wherein the sum of each component content is 100wt%, and each weight percentage is based on the

total weight of the amine curable epoxy resin composition.

2. The amine curable epoxy resin composition according to claim 1, wherein the content of N,N'-bis(3-aminomethylbenzyl)-2-hydroxytrimethylenediamine is 72-76wt% of the first amine based composition and the content of m-xylylenediamine is 24-28wt% of the first amine based composition in the first amine based composition.

3. The amine curable epoxy resin composition according to claim 1 or 2, wherein the content of m-xylylenediamine is less than 1wt% of the second amine based composition in the second amine based composition.

4. The amine curable epoxy resin composition according to claim 1, further comprises silica sand.

5. The amine curable epoxy resin composition according to claim 1, wherein the hardener component further comprises benzyl alcohol.

6. The amine curable epoxy resin composition according to claim 1, wherein the alkyl in the alkylsulphonic phenyl ester is C12 to C18.

7. The amine curable epoxy resin composition according to claim 6, wherein the alkylsulphonic phenyl ester is dodecylsulphonic phenyl ester.

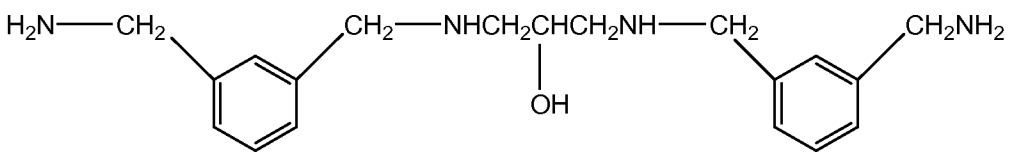
8. The amine curable epoxy resin composition according to claim 1, wherein the wetting and dispersing agent is a solution of copolymer with acidic groups.

9. A hardener comprising:

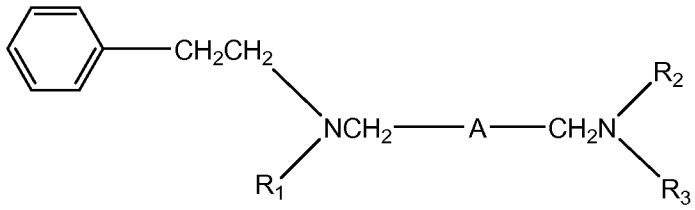
20-90 wt% of first amine based composition, and

10-80 wt% of second amine based composition,

wherein the first amine based composition comprises N,N'-bis(3-aminomethylbenzyl)-2-hydroxytrimethylenediamine represented by the following formula (I) and m-xylylenediamine, and the second amine based composition comprises the reaction products of m-xylylenediamine and styrene represented by the following formula (II) and m-xylylenediamine,



(I)



(II)

wherein A is phenylene, and each of R_1, R_2 and R_3 independently represents hydrogen or phenylethyl;

wherein the sum of each component content is 100wt%, and each weight percentage is based on the total weight of the first amine based composition and the second amine based composition.

10. The amine curable epoxy resin composition according to claim 9, wherein the content of N,N'-bis(3-aminomethylbenzyl)-2-hydroxytrimethylenediamine is 72-76wt% of the first amine based composition and the content of m-xylylenediamine is 24-28wt% of the first amine based composition in the first amine based composition.

11. The amine curable epoxy resin composition according to claim 9 or 10, wherein the content of m-xylylenediamine is less than 1wt% of the second amine based composition in the second amine based composition.

12. The use of the amine curable epoxy resin composition according to claim 1 as primer, topcoat and screeds for application on concrete.

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2013/053762

A. CLASSIFICATION OF SUBJECT MATTER
INV. C08G59/22 C08G59/50 C08L63/00
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
C08G C08L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	GB 2 286 592 A (SANDOZ LTD [CH] SANDOZ LTD [CH]; MBT HOLDING AG [CH]) 23 August 1995 (1995-08-23) cited in the application page 16; example 5 claims 1-10	1-12
A	WO 98/17724 A1 (HENKEL CORP [US]) 30 April 1998 (1998-04-30) claims 1-22 page 21; example 8	1-12
A	EP 0 088 623 A1 (MITSUBISHI GAS CHEMICAL CO [JP]) 14 September 1983 (1983-09-14) claims 1-8 pages 10-11; example Ref. ex. 1	1-12

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[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

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Date of the actual completion of the international search	Date of mailing of the international search report
16 August 2013	23/08/2013

Name and mailing address of the ISA/	Authorized officer
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INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2013/053762

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

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A	WO 2005/123800 A1 (HUNTSMAN ADV MAT SWITZERLAND [CH]; HUNTSMAN ADVANCED MATERIALS DE [DE]) 29 December 2005 (2005-12-29) claims 1-11 Table; page 6	1-12

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