ANTICORROSION TAR EPOXY COATINGS

Abbas H. Sulaymon", Mooaid F. Al-Muddaris and Ali A. Merdaw

Chemical Engineering Department – College of Engineering – University of Baghdad – Iraq Environmental Engineering Department for Higher Studies– College of Engineering – University of Baghdad – Iraq

ABSTRACT

The present study refers to a tar-epoxy resin coating formulations consisting mainly of blowing asphalt, solvent (xylene), epoxy resin (EPON 828) and curing agent (DETA). These materials are made into two components, (A) and (B). When (A) and (B) are mixed in a specified mixing ratios and painted upon steel structure surface, a strong anti-corrosive coating layer is formed. Tar-epoxy paints are widely applied to surface anti-corrosive project of various structures in different environments.

Key words: Epoxy / coatings / tar-epoxy coatings

INTRODUCTION

Epoxy surface coatings are among the most widely used industrial finishes, exceeded in volume only by alkyds and acrylics. Epoxies are often more expensive than other coatings, but provide superior adhesion, flexibility and corrosion resistance when applied to metallic substrates⁽¹⁾.

While the cost of corrosion to mankind is extremely difficult to measure, some experts estimate that over \$100 billion are lost annually in the United States alone through the effects of corrosion. In addition to damage costs, corrosion has prompted some engineers to over design structures by specifying extra metal thickness for additional life and strength. As a result, lacking effective corrosion control, countless tons of metal are consumed needlessly ⁽²⁾.

The aim from this study is introducing new formulations for tar epoxy coatings produced by using locally industrial products, such as asphalt (blowing or paving) and an aromatic hydrocarbon solvent (xylene). Tar epoxy coatings is a high build two pack water proofing and anticorrosive system based upon epoxy resin, tar and amine curing agents specially chosen for their ability to withstand high degrees of corrosion and also for mild chemical attacks

Several patents investigate different formulations for the tar epoxy coatings and its applications⁽³⁻²³⁾. Several of these patents were useful and were helpful to the experimental work in this study.

Whittler and Lawn⁽⁷⁾ prepared four corrosion resistant bituminous coating formulations especially adapted for application to steel and concrete surfaces, consisting of DGEBA epoxy resin, DETA curing agent, aromatic solvent (high flash naphtha, xylene or toluene), coal tar pitch and inorganic filler (talk) in different weight ratios.

Several formulations had been prepared by Lopata⁽⁸⁾ using DGEBA epoxy resin, DETA or PA as a curing agents, middle oil (distilled from coal tar in the temperature range of 200 to 320°C) and finely divided silica, clay and/or carbon as fillers and xylene as a solvent, in different weights.

Simpcon⁽⁹⁾ had tested several formulations consisting of epoxy resin (DGEBA, diglycidyl ether of resorcinol or diglycidyl ether of 2,2-bis(4hydroxyphenyl)butane), DETA curing agent and petroleum residue, to form coatings cured at atmospheric temperature.

Another experiments carried out by Herzberg⁽¹²⁾ using Epoxy resin obtained by epichlorohydrin with condensing bis-(4hydroxyphenyl)-dimethyl methane. This resin was incorporated with coal-tar pitch (softening temperature 90°C), distillation bitumen (softening temperature 85°C), and blown bitumen (softening temp. 87°C) in different concentrations. Several solvents had been used: o-dichlorobenzene, methyl isobutyl ketone, toluene, etc. The curing agent used in this experiment was ureaformaldehyde resin.

A road surfacing composition, paving composition and a process for treating surfaces to render them non-skid were suggested by Wittenwyler⁽¹³⁾, using a products derived from coal of the residual group consisting of coal-tars, refined coal-tars and coal-tar pitches having a softening point below 88 °C. The coal product and polyepoxide had been composed in weight ratios ranging from 15:1 to 1:15.

Boenau, Bruins and Salvesen ⁽¹⁴⁾ made many samples for tar-epoxy coatings. Chemical resistance and electrical properties had been measured for each formulation sample. Formulations were differ in the aromatic hydrocarbon modifiers and curing agent's concentrations.

EXPERMANTAL WORK

Surface preparation

For all epoxy coatings, the best performance results are obtained by blasting metal surfaces to near-white or white metal. These surface finishes can be achieved by using commonly available abrasives such as sand, steel grit, aluminum oxide, garnet, etc. The most common blast grade specified is a near-white surface finish, which allows very light shadows or slight discoloration of the metal ^(24,25,26).

Chemical materials used Epoxy resins

The epoxy resin, which had been used in this study, is EPON 828, produced by Shell Company. EPON 828 is an undiluted clear difunctional DGEBA derived epoxy resin. Specifications of this material are listed in table (1) ⁽²⁷⁾.

Curing agents

The curing agent, which had been used in this study, is DETA (diethylenetriamine), produced by Fluka Chemie AG (Switzerland) as a laboratory reagent (assay > 97%). DETA used as a curing agent for DGEBA resins in amounts between 8-12 phr. In this study, at first, a concentration of 12 phr had been used as recommended in the data sheet for EPON resin 828. Table (2) indicates the specifications of DETA.

Modifying additives Diluents

The only diluent material, which had been used in this study, is xylene. Table (3) indicates its specifications.

Resinous modifiers

Two kinds of asphalt had been used: blown asphalt and paving asphalt, both are produced industrially in Iraq by the Al-Dura refinery. Tables (4) and (5) indicates the specifications of these materials.

Table (1): Specifications of The Epoxy Resin

Specifications	Value
Epoxide equivalent weight	185-192
Viscosity, at 25 °C, poise	110-150
Color, Gardner	1 max
Physical form	Clear liquid
Pounds/gallon at 25 °C	9.7
Density, g/ml at 25 °C	1.16
Flash point, °F	No flash at 249 °C
Vapor pressure, mm Hg at 77 °C	0.03
Refractive index at 25 °C	1.573
Specific heat, BTU/lb/°F	0.5

Table (2): Specifications of DETA (28)

Specifications	Value	
Formulation	NH2C2H4NHC2H4NH2	
Molecular weight	103.17	
Boiling point, °C	200~205	
Fusion point, °C	-39	
Specific gravity (20/20°C)	0.9542	
Vapor pressure (20°C), mm	0.37	
Flash point, °C	101.6	
Viscosity (20°C), poise	0.0714	
Coefficient of expansion	0.00088	

Table (.	3): S	pecij	fications	of	Xylene
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Specifications	Value
Formulation,(dimethylbenzene)	$C_{6}H_{4}(CH_{3})_{2}$
Molecular weight	106.17
Grade	General purpose reagent
Boiling range (95%), °C	137~142
Weight per ml at 20°C	0.855~0.865 g
Maximum limits of impurities;	
Non-volatile matter	0.01%
Sulfur compounds (CS ₂)	0.0003%
Solution in alcohol	Clear

Specifications	Value	
Grade	20~30	
Penetration at 25 °C (100 g, 5 sec, 0.1mm)	20~30	
Softening point (R&B), °C	80~90	
Specific gravity at 15.6°C	1.0~1.1	
Flash point (C.O.C.), °C minimum	240	
Ductility at 25°C, (cm) minimum	3	
Solubility in CCl ₄ , %wt. minimum	99	
Loss on heating, %wt. maximum (5 hrs.,163 °C)	0.2	

Table (4): Specifications of Blown Asphalt

Table	(5)	: Speci	fications	of Paving	Asphalt

Specifications	Value
Grade	40~50
Penetration at 25 °C (100 g, 5 sec, 0.1 mm)	40~50
Softening point (R&B), °C	49~58
Specific gravity at 15.6°C	1.04
Flash point (C.O.C.), °C minimum	240
Ductility at 25°C, (cm) minimum	100
Solubility in CCl ₄ , %wt. minimum	99
Loss on heating, %wt. maximum (5 hrs.,163 °C)	0.5
Penetration of residue after loss on heating, % of the original value (minimum)	75

The experiments

Several literatures and patents ^(7-9,12-14,29,30) had been studied to get a guide to choose a tar epoxy coating formulations.

In order to choose the better formulation for this product, the experiments were divided into two categories, the first is by using blowing asphalt and the second is by using paving asphalt. Both kinds of asphalt were incorporated in different concentrations with the same other materials (epoxy resin, curing agent and diluent).

Each formulation contains two main components (A and B). (A) Component, which include epoxy resin (referred by R), diluent (referred by SA) and blowing asphalt (referred by L) or paving asphalt (referred by V), and (B) component, which include the curing agent (referred by C) and diluent (referred by SB).

Each formulation's sample had been prepared and then coated manually (by brush) in a single buildup, on a prepared metal and glass flat plates (1 x 3in.). Coated films are then cured utilizing one of two curing schedules;

a. At room temperature (30°C) for 7 days.

b. At room temperature $(30^{\circ}C)$ for 6 hrs., then at $100^{\circ}C$ for 1 hr.

Tests were carried out directly after the curing schedules. These tests included; the visual inspection, measuring the thickness, measuring the hardness and immersion in water to find the water absorption resistance, of the coated film.

Samples Preparation

The component (A) was prepared by mechanical blending of the three materials R, SA and L or V, in a glass or metal container for 30 minutes with heating to 65° C, by using a hot plate magnetic stirrer (M6, Witeg Electric, Germany). In the same manner, component (B) was prepared from its components C and SB, but without heating.

Each component, (A) and (B) were kept in separated containers. Before application on surface, the two components were mixed according to its calculated mixing ratio in a disposable flasks for at least 3 minutes to insure good blending, then the mixtures were left for 1 hour (induction period), to permit the polymerization reaction to initiates and propagate. Effect of Asphalt

Table (6) indicates the composition of each formulation's sample in a first set of experiments. In this set of experiments a constant concentration of curing agent (DETA) had been used (12 phr). Each formulation's sample undergoes several tests:

Measuring The Density

The density of each sample had been calculated by dividing sample weight by its own volume at room temperature (30°C) by using a 50-milliliter pycnometer. Results of density and asphalt concentration for each sample are listed in table (7).

Hardness Test

Hardness of the coated film had been measured by Vickers micro hardness method according to ASTM E384 and by utilizing a micro hardness tester (Micromet, Adolph I. Buehler Inc., USA) for each sample. A measuring microscope mounted on the machine in such a manner that the indentation may be readily located in the field of view.

The Vickers hardness numbers are given in special tables⁽³¹⁾ for test loads (gf) and diagonal impression (μ m). In this study the hardness values are obtained directly from this special tables.

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Table (8) shows the results of the hardness test, the subscript letter under the number of sample is referring to the curing schedule (a) or (b).

Film thickness for the coating samples were varying between 0.15 mm for sample no.1 to 0.35 mm for samples no.5 and no.9.

<i>Table</i> (6):	Formulations	of The	First Set	of Experiments

Formulation Sample	(A) Component, ppw.			(B) Component, ppw		Mixing Ratio A:B	
	R	SA	L	V	C	SE	(weights)
1	100	20	0	0	12	8	6.1
2	100	30	50	0	12	8	9:1
3	100	40	100	0	12	8	12.1
4	100	50	150	0	12	8	15:1
5	100	60	200	0	12	8	18.1
6	100	30	0	50	12	8	9.1
7	100	40	0	100	12	8	12.1
8	100	50	0	150	12	8	15.1
9	100	60	0	200	12	8	18.1
	lt, C: (weight	Curing	agent (D				: Blown asphalt, l component (xylene

Table (7): First Set Samples Densities

Sample	Density at	30°C, g/mi	Asphalt
	A comp.	B comp.	concentration, phr
1	1.12	0.93	0
2 1	1.10	0.93	50
3	1.07	0.93	100
A Blowing	1.044	0.93	150
5 1	1.04	0.93	200
67	1.08	0.93	50
7	1.06	0.93	100
8 Paving	1.04	0.93	150
Le	1.035	0.93	200

Table (8): Hardness Test Results for The First Set of Experiments.

Sample	Asphalt, phr	HV*	Notes
1.	0	21.950	OK
16	0	25.850	OK
2.7	50	6.16010	Slight surface cloudiness
20	50	6.19610	Slight surface cloudiness
3.	100	6.01610	Slight surface cloudiness
36	100	5.73410	Slight surface cloudiness
4. Blowing	150	no reading	Soft coated film
4. Asphalt	150	no reading	Soft coated film
5.	200	no reading	Very soft coated film
5, 1	200	no reading	Very soft coated film
6.7	50	6.56310	Slight surface cloudiness
65	50	7.72710	Slight surface cloudiness
7.	100	no reading	Soft coated film
76	100	no reading	Soft coated film
8. Paving	150	no reading	Very soft coated film
8 Asphalt	150	no reading	Very soft coated film
9.	200	no reading	Very soft coated film
5° 7	200	no reading	Very soft coated film

Water Absorption Resistance

The samples were tested for 2 hours immersion in boiling water, according to ASTM D 570.

The test specimen was in the form of sheet, 76.2 mm (3 in.) long by 25.4 mm (1 in.) wide by the thickness of the material. Coated film sheets were applied on glass plates, in a single buildup. After curing by utilizing one of the curing schedules (a) or (b), this film sheets were removed from the glass plates by the aid of a special release agent (polyvinyl alcohol 5% in water) had been previously coated and dried on the glass plates.

The specimen placed in a container of boiling distilled water, supported on edges and had been entirely immersed. At end of 120 ± 4 min, the specimens removed from water and cooled in distilled water maintained at room temperature. After 15 ± 1 min, the specimen removed from water, one at a time, all surface water removed with a dry cloth, and the specimens weighed to the nearest 0.000001 g immediately.

Results for the test of two hours boiling water immersion are indicated in table (9).

Percentage increase in weight during immersion, was calculated to the nearest 0.01 % as follows:

Increase in weight, $\% = 100 \times \frac{(wet wt. initial wt.)}{(initial wt.)}$

Sample	Asphalt, phr	Increase in weight*, %	Visual notes [§]
1 _a	0	0.26	OK
16	0	0.07	OK
2.7	50	0.29	OK
2.	50	0.95	OK
3.	100	0.13	1L
36	100	2.86	1L
4. Blowing	150	and and the second second	1D
4 Asphalt	150		1D
5.	200		3D.3C
5, 1	200	the strategiest of the	3D,3C
6.7	50	0.58	OK
65	50	1.56	OK
7.	100	0.71	1B.1D
76	100	0.82	1B.1D
8. Paving	150	Contraction of the second second	1D,1C
86 Asphalt	150	(URANA) ARAAN ARA	1D.1C
9.	200	Estevicity 1 1 / 2	3D,3C
r se	200		3D.3C

Table (9): Immersion Test for The First Set of Experiments.

Effect of Curing Agent Concentration

Examining tables (15) and (16), it was found that sample no.2 and sample no.3 were having the better results. Table (10) indicates a new set of formulation samples. The formulation's samples no.3 and no.2 had been chosen to be the reference to the second set of experiments (table 10), therefore, component (A) in the formulation's samples (10, 11, 12 and 13) was the same as in sample no.3 (table 6), while component (A) in the other formulation samples (14, 15, 16 and 17) was the same as in sample no.2 (table 6). Component (B) had been changed by reducing the curing agent (C) concentration and increasing the diluent concentration (SB) to maintain a constant mixing ratio of the two components (A) and (B).

Using the same procedure of the first set, samples in this second set had prepared and tested. Results are listed in table (11) for hardness test and in table (12) for immersion test.

Table (10): Formulations of The Second Set of Experiments

eights)
2:1
2:1
2:1
2:1
9:1
9.1
91
9:1
9:1

Table (11): Hardness	fest Results for The Se	econd
Set of Experiments		

Sample	Hardener, phr	HV*	Notes
10.	8	4.505	OK
100	8	4.50s	OK
11.	9	5.5010	OK
116	9	5.6510	OK
12.	10	5.56210	OK
120	10	5.93010	OK
13.	11	5.88010	Slight surface cloudiness
136	11	6.02510	Slight surface cloudiness
3.	12	6.01610	Slight surface cloudiness
36	12	5.73410	Slight surface cloudiness
14,	8	5.72310	OK
140	8	5.85310	OK
15a	9	5.66010	OK
150	9	5.98610	OK
16.	10	6.12510	OK
160	10	6.34210	OK
17.	11	6.05510	OK
176	11	6.26810	OK
2.	12	6.16010	Slight surface cloudines:
26	12	6.19610	Slight surface cloudiness

Table (12): Immersion Test for The Second Set of Experiments

Sample	Hardener, phr	Increase in weight, %	Visual notes§
10.	8	+ 0.53	1T
100	8	+ 1.99	1T
11.	9	+ 0.68	OK
116	9	+ 2.12	OK
12.	10	+ 0.66	OK
126	10	+ 2.23	OK
3.	11	+ 0.59	OK
30	11	+ 2.56	OK
3.	12	+ 0.13	1L
36	12	+ 2.86	1L
4.	8	+ 0.48	OK
40	8	+ 0.55	OK
5a	9	+ 0.45	OK
50	9	+ 0.68	OK
6.	10	+ 0.35	OK
60	10	+ 1.11	OK
7.	11	+ 0.64	OK
70	11	+ 0.89	OK
2.	12	+ 0.29	OK
20	12	+ 0.95	OK
	water otes after imme B. Blister	red before and after 2 ho (ASTM D 570). Astron: 1. Slight, 2. Moder ing, T. Tacky, C. Discolor OK. Unchanged, R. Rus	ate, 3. Severe. ation, D. Dissolving,

Long-Term Immersion

Final long-term immersion tests had been carried out using two formulation's samples selected from the second set of experiments.

Long-term immersion test had been carried out according to ASTM D570. Immersion achieved in distilled water at 28°C. Long-term immersion test results are listed in table (13).

Table (13): Long-Term Immersion Test Results.

Time,		% Increase in v	weight with formu	lation.
day	no.11.	no.11b	no.15.	no.150
0	0	0	0	0
1	0.04	0.10	0.03	0.04
7	0.35	1.01	0.22	0.36
14	0.42	1.20	0.28	0.44
28	0.43	1.34	0.29	0.45
42	0.46	1.35	0.32	0.50
56	0.48	1.44	0.35	0.53
70	0.49	1.45	0.36	0.53
84	0.50	1.53	0.38	0.54
98	0.51	1.54	0.40	0.55

Finally, table (14) gives a specifications summary of the two selected samples (11 and 15).

Pot life measured by using a rotary viscometer (Haake, Germany). Pot life generally ends at a viscosity of about 5000 centipoises^[29].

Spread rate had been measured by estimating the areal density of the cured paint. A flat metal plate (40x40cm) coated with 0.2mm thick layer, was used for this purpose. Table (14): Summary of The Selected Formulations Specifications.

Ingredients	Concentration, ppw		
	Formulation no.11		
Component A:			
Epoxy resin (Epop 828)	100	100	
Blowing asphalt	100	50	
Xvlene	40	30	
Component B	~	20	
DETA	0	0	
Xylene	11	11	
Specifications	Value		
Pot life, 200 g at 30°C, minutes	90	75	
Hardness, Vickers,			
Curing 7 days at 30°C	5.5010	5.66010	
Curing: 1 hr. at 100°C	5.6510	5.98610	
Distilled water absorption, %	10		
After 2 hrs. boiling		1	
Curing 7 days at 30°C	+0.68	+0.45	
Curing 1 hr at 100°C	+2.12	+ 0.68	
After 14 weeks at 28°C		. 0.00	
Curing: 7 days at 30°C	+0.51	+0.40	
Curing: 1 hr. at 100°C	+1.54	+0.55	
Spread rate on metal surfaces:		10.55	
m ² /Kg at 0.2 mm thick	25	34	
m ² /1 at 0.2 mm thick	27	37	
Specific gravity at 30°C:			
Component (A)	1.07	1.10	
Component (B)	0.93	0.93	
Mixture	1.06	1.09	
Mixing ratio (A:B):	and the second second		
by weights	12:1	9:1	
by volumes	10.41	7.6:1	

RESULTS AND DISCUSSION Effect of Asphalt

Two kinds of asphalt had been used in this study: blowing asphalt (table4) and paving asphalt (table 5). Several suggested formulations (table 6) incorporate these asphalts with the other chemical materials: epoxy resin (EPON 828), solvent (xylene) and curing agent (DETA). Curing agent concentration was constant (12 phr) in each formulation, according to the EPON 828 product bulletin. Solvent concentration increased proportionally with increasing of asphalt concentration in the formulation, to get better blending viscosity while preparation.

Density of component (A) in each formulation had been measured (table 7) and plotted against asphalt concentration in figure (1). Densities of component (A) were decreasing where is the asphalt and diluent concentrations increases.

Results of hardness testing (table 8) for cured samples utilizing cure schedules (a) and (b) had been plotted in figure (2), against asphalt concentration. Results of immersion test (table 9) were plotted in figure (3).

As the results of the first set of experiments indicates, the following major notes had been found:

- 1. Curing agent (DETA) concentration of 12 phr was high, and result a slight surface cloudiness in many samples.
- Blowing asphalt is better than paving asphalt in the tar epoxy coatings, due to its higher softening point. Many samples incorporate

paving asphalt had no hardness reading, because of the softness of the coated film were to a degree that the hardness tester could not measure it.

- Utilizing curing schedule (b) gives higher hardness values than the curing schedule (a).
- Utilizing curing schedule (b) gives all samples incorporated asphalt in composition lower resistance to water absorption than utilizing curing schedule (a). But gives the opposite effect on samples that had no asphalt in the composition.
- 5. Increasing asphalt concentration had a lowering effect on the sample resistance to water absorption.
- 6. Better blowing asphalt concentration was in samples 2 and 3, were the asphalt concentration 50phr & 100phr respectively, in both tests hardness and water absorption resistance.

Effect of Curing Agent Concentration

Samples no.2 (50phr blowing asphalt) and no.3 (100phr blown asphalt) had been chosen to be the reference to this set of experiments.

Various concentrations of curing agent (DETA) had been examined, between 8phr to 12phr (table 10).

Results of hardness test (table 11) plotted in figure (4), while figures (5) shows the results of immersion test (table 12).

A review to the results collection above indicates that:

- 1. Better hardness results with stoichiometric concentration of curing agent (10~11 phr).
- 2. Better water absorption resistance results with minimum concentration of curing agent (8 phr).
- 3. Formulations with asphalt concentration of 50phr were better than formulations with 100 phr in each hardness and immersion tests, i.e. it had better specifications.
- 4. Formulations with asphalt concentration of 50phr had a higher cost than formulations with 100phr.

Samples no.11 and no.15 had been regarded as the best formulations, where is the low curing agent concentration (9 phr) gives good water absorption resistance, and quite near to the stoichiometric concentration to get a reasonable hardness values.

Long-Term Immersion

Long-term immersion test at room temperature (28°C) in distilled water had been carried out (table 13). Results of this test plotted in figure (6).

This long-term immersion test results shows that:

- 1. Formulation's sample no.15 (50phr asphalt) had better resistance to water absorption than formulation's sample no.11 (100phr asphalt).
- 2. Utilizing curing schedule (a) gives better water absorption resistance than schedule (b).

CONCLUSIONS

- a. New formulations had been prepared in this study for the tar-epoxy coatings consisting of some locally produced technical materials, such as blowing asphalt and xylene.
- b. Tow types of asphalt experimented; blowing asphalt of grade (20-30), and paving asphalt of grade (40-50) to be used in tar epoxy coatings. Experiments improves that the first type is better than the second, due to its higher softening point.
- c. Hardness value of the coated film increases with lowering the asphalt concentration in the formulation.
- d. Better hardness values of the coated film were measured near the stoichiometric concentration of the curing agent (10~11 phr DETA).







Figure(2) Vickers hardness for the first set formulas vs asphalt concentration

- e. Utilizing the curing schedule (b): one hour at 100oC, gives better values of hardness than utilizing the curing schedule (a): seven days at 30oC. That is due to a higher cross-linking density obtained by schedule (b) than schedule (a).
- f. Immersion tests in distilled boiled water improves that increasing the asphalt concentrations gives lower water absorption resistance for the coated films.
- g. Immersion tests in boiled distilled water for two hours improve that reducing curing agent concentration than stoichiometric gives better water absorption resistance.
- h. Utilizing curing schedule (a) gives better water absorption resistance than utilizing curing schedule (b) for the coated film.
- i. Results of long-term immersion in distilled water at 28°C for about 3 months shows the same effects of materials concentrations and curing schedule type on water absorption resistance mentioned above, but always with lower weight gains.





Figure (3) Immersion test for the first set of experiments

Figure (4): Hardness results for the second set of experiments

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Figure (5) Immersion test results for the second set of experiments



Figure (6): Long-term immersion test (distilled water at 28 °C)

Nomenclature

ASTM	American Society for Testing
and Materials	
DETA	diethylenetriamine
DGEBA	diglycidyl ether bisphenol A
PA	phthalic anhydride
phr	parts per hundred parts of resin

Symbols

C	curing agent (DETA)
L	blowing asphalt
R	epoxy resin (EPON 828)
SA	diluent of component (A)
S _B V	diluent of component (B)
V	paving asphalt

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