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(54) **COMPOSITIONS CHIMIQUES**

(54) **CHEMICAL COMPOSITIONS**

(57) Système en emballage groupé comprenant au moins deux composants qui réagissent ensemble pour former une résine thermo-durcissante, caractérisé en ce qu'un des composants contient jusqu'à 0,01 % en poids d'un indicateur de pH.

(57) A multi-pack system which comprises two or more components which react together to form a thermosetting resin characterised in that one of the components contains up to 0.01 % by weight of a pH indicator.

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(57) Abstract

A multi–pack system which comprises two or more components which react together to form a thermosetting resin characterised in that one of the components contains up to 0.01 % by weight of a pH indicator.

Chemical Compositions

The present invention relates to multicomponent liquid systems which react to produce such products as thermosetting resins.

Thermosetting resins such as polyurethanes and epoxy resins are widely used, e.g. as adhesives, for making coatings and in tooling applications. In many cases the mixture of the components is very similar in appearance to some or all of the individual components. It is therefore possible to produce products where one or more components are missing, e.g. due to machine breakdown, and for the operator to be unaware of such an occurrence.

Sometimes a dye or pigment is added to a component and it is then possible to ensure proper mixing by observing the colour of the mixture, and comparing it to the colour of the individual component. This normally requires 0.1 to 5% of costly dyestuff or pigment.

We have now surprisingly found that a very small amount of a compound which is normally used as an indicator of pH change, also exhibits a change in colour in the thermosetting systems.

Accordingly the present invention provides a multi-pack system which comprises two or more reactive components which react together to form a thermosetting resin characterised in that one of the reactive components contains up to 0.01% by weight of a pH indicator.

Usually the system of the invention will comprise two packs, each of which contains a reactive component.

For example, in the case of polyurethanes, one part will comprise a polyol and the second will comprise a diisocyanate. In the case of epoxides, one part will comprise an epoxy resin and the second will comprise a hardener.

Only very small amounts of indicator are needed to produce a desired colour change. Usually less than 0.01% by weight of the component to which it is added is sufficient. Preferably the amount is from 0.0003% to 0.006%, and more preferably from 0.0005% to 0.004%.

The indicator may be added to any component provided that it does not react with that component and thereby prevent the colour change.

For instance in the case of polyurethanes, many indicators, such as those which contain one or more phenolic groups, will react with isocyanates thereby destroying the chromophore. In this case the indicator should be added to the polyol component.

Similarly in the case of epoxy systems, amines or amine catalysts in the hardener will react with phenolic groups in the indicator which is therefore added to the epoxy component. In the case of high temperature phenolic cured epoxides the indicator may be added to the phenolic hardener.

Any indicator which produces a change in colour when the components are mixed may be used. The indicator however is preferably one which, in aqueous systems, at least begins to change colour at a pH below 8.2. Those that are either strongly coloured in both acid and alkaline states or those that give a strong alkaline colour at low pH values, e.g. below 5.0 are especially preferred. As examples of suitable indicators, there may be mentioned Cresol Red, Bromophenol Blue, Bromocresol Green, Bromocresol Purple, Chlorophenol Red, Phenol Red and Thymol Blue.

It should be noted that the colour change observed is not always the same in different systems and is also not always the same as the colour change produced on changing the pH in an aqueous system. For example, Chlorophenol Red changes from orange to beige in polyurethane systems and from yellow/beige to purple in epoxies. When indicating a pH change the colour change is from yellow to red.

When mixing the two components, the person carrying out the operation can easily see from the colour change that the two components have indeed been mixed, and that the mixing is even throughout the mixture.

The invention is illustrated by the following Examples.

Examples 1-6

Polyurethanes are prepared from a polyol component (A) and component (B) which is semi-distilled liquid 4,4'-methylene diphenyl diisocyanate (MDI) in a ratio of (A):(B) of 100:25 by weight. Component (A) has the composition:-

Polyol Component - (A)

	%w/w
Castor Oil BP	32.1
Branched Polyether Polyol - Equivalent Weight 148 (Desmophen 55OU, available from Bayer)	13.9
Dolomite	49.0
Fumed Silica	2.0
Molecular Sieve	3.0

Indicators are added to component (A) as a 0.1% solution in a suitable carrier which is castor oil or a polyol (Lupranol 1000, BASF). The following results are obtained.

Example	Indicator	%w/w on Component (A)	Colour change on mixing with Component (B)
1	Bromophenol Blue	0.003	Blue → Beige
2	Bromocresol Green	0.003	Green → Beige
3	Bromocresol Purple	0.003	Yellow → Beige
4	Cresol Red	0.003	Light Yellow → Beige

5	Chlorophenyl Red	0.003	Orange → Beige
6	Phenol Red	0.003	Light Pink → Beige

Examples 7-11

Cured epoxy resins are produced from epoxy resin component (A) and curative component (B) in a mixing ratio (A):(B) of 100:37 by weight. Component (A) has the composition:-

Epoxy Resin Component - (A)

	%w/w
Epichlorohydrin Bisphenol A Epoxy Resin	31.71
Glycidyl Ether of a C_{12} - C_{14} Alcohol	12.52
Dibutyl Phthalate	4.15
Sorbitan Monoisostearate	0.35
Calcium Carbonate	49.72
Fumed Silica	1.21
Titanium Dioxide	0.34

Component (B) has the composition:-

Curative Component - (B)	%w/w
Amine Accelerated Polyaminoamide	70.00
Tris Dimethylaminomethyl Phenol	10.00
Benzyl Alcohol	20.00

Indicators are added to component (A) as a 0.1% solution in polyol (Lupranol 1000). The following results are obtained.

Example	Indicator	%w/w on Component (A)	Colour change on mixing with Component (B)
7	Phenol Red	0.003	Beige → Pink
8	Cresol Red	0.003	Beige → Light Purple
9	Bromocresol Purple	0.003	Yellow/Beige → Violet
10	Thymol Blue	0.003	Yellow/Beige → Grey/ Beige
11	Chlorophenol Red	0.003	Yellow/Beige → Purple

Examples 12-14

Following the procedure of Examples 1-6 where the ingredients are as given below, MDI being component (B) and the remainder are component (A), the colour change obtained on adding (B) to (A) is as stated.

Example	12	13	14
N,N,N^{i},N^{l} - Tetrakis (2-Hydroxypropyl) Ethylene Diamine (Quadrol "L", BASF)	30	-	-
Branched Polyether Polyol Equivalent Weight 148 (Desmophen 550U, Bayer)	-	44	-
Castor Oil No. 1	-	-	44
Highly Reactive Polyether Triol (Lupranol 2042, BASF)	64	-	-
Dolomite	-	-	50

Calcium Carbonate	-	50	-
Molecular Sieve	3	3	3
Thymol Blue (0.1% in Lupranol 1000)	3	-	-
Bromophenol Blue (0.1% in Lupranol 1000)	-	3	-
Bromocresol Green (0.1% in Lupranol 1000)	-	-	3
Modified MDI (Lupranat MP102, BASF)	80.5	-	-
Semi-Distilled MDI (Lupranat M20S, BASF)	-	40	-
Semi-Distilled MDI (Desmodur 44V20L, Bayer)	-	-	17
Colour Change	Yellow - Cream/White	Blue - Beige	Grey/Green - Beige

Examples 15-18

Different amounts of indicator are used in one polyurethane system to show the different colour changes. The ingredients used and the colour changes are as follows

Example	15	16	17	18
N,N,N^{l}N^{l}-Tetrakis(2-Hydroxy Propyl) Ethylene Diamine (Quadrol "L", BASF)	30	30	30	30
Highly Reactive Polyether Triol (Lupranol 2042, BASF)	64	64	64	64
Molecular Sieve	3	3	3	3
Bromophenol Blue	0.006	0.001	0.0005	0.0003
Semi-Distilled MDI (Desmodur 44V20L, Bayer)	59	59	59	59

Colour Change on adding MDI	From	Deep Blue	→	Very Light Blue
	To	Light Blue	→	Colourless

Claims

1. A multi-pack system which comprises two or more components which react together to form a thermosetting resin characterised in that one of the components contains up to 0.01% by weight of a pH indicator.

2. A system as claimed in claim 1 in which the amount of indicator is from 0.003% to 0.006% by weight.

3. A system as claimed in claim 1 or 2 in which the indicator is one which, in aqueous systems, at least begins to change colour at a pH below 8.2

4. A system as claimed in any preceding claim in which the indicator is one which is strongly coloured in both acid and alkaline states.

5. A system as claimed in any one of claims 1 to 3 in which the indicator is one which gives a strong alkaline colour at pH values below 5.0.

6. A system as claimed in any preceding claim in which the indicator is Cresol Red, Bromophenol Blue, Bromocresol Green, Bromocresol Purple, Chlorophenol Red, Phenol Red or Thymol Blue.

7. A system as claimed in any preceding claim which comprises a polyol as one component and an isocyanate as a second component and the indicator is present in the polyol component.

8. A system as claimed in any one of claims 1 to 6 which comprises an epoxy resin as one component and a hardener as a second component and the indicator is present in the epoxy resin component.

9. A system as claimed in claim 1 substantially as hereinbefore described with reference to any one of the foregoing Examples.