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Bezeichnung der Erfindung: Thermosetting binders, preparation and use in lacquers
Title of invention:
Titre de l'invention :

Klassifikation / Classification / Classement : C08G 59/14

ENTSCHEIDUNG / DECISION

vom / of / du 1 December 1987

Anmelder / Applicant / Demandeur : Shell International Research Maatschappig B.V.

Patentinhaber / Proprietor of the patent / Titulaire du brevet :

Einsprechender / Opponent / Opposant :

Stichwort / Headword / Référence : Thermosetting binders/Shell

EPÜ / EPC / CBE Article 56

Kennwort / Keyword / Mot clé : "Inventive step"

Leitsatz / Headnote / Sommaire

Case Number : T 47/85

DECISION
of the Technical Board of Appeal 3.3.1
of 1 December 1987

Appellant : Shell Internationale Research
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Decision under appeal : Decision of Examining Division 012 of the European Patent Office dated 29 August 1984 refusing European patent application No. 81 200 009.9 pursuant to Article 97(1) EPC

Composition of the Board :

Chairman : K.Jahn
Members : R. Andrews
R. Schulte

Summary of Facts and Submissions

I. European patent application No. 81 200 009.9, filed on 6 January 1981 and published on 5 August 1981 (publication No. 0 033 169) claiming priority of 25 January 1980 from a prior application filed in the United Kingdom, was refused by a decision of the Examining Division 012 of the European Patent Office dated 29 August 1984. The decision was based on Claims 1 to 11 filed on 19 may 1983. Claim 1 reads as follows.

"A process for the preparation of thermosetting binders which are water-soluble when neutralised with a base, characterised in that a polyglycidyl ether of a polyhydric phenol is reacted with an aliphatic amino acid in which at least 50% of the acid groups have been neutralised with an alkali metal hydroxide, a tertiary amine or a quaternary ammonium hydroxide, in a ratio of 0.7 to 1.2 aminohydrogen equivalent per epoxy equivalent, reaction being initiated by heating to 90 to 100°C".

II. The stated ground for the refusal was that the subject-matter of Claim 1 did not involve an inventive step. The process represented an obvious alternative to the one described in US-A-4 098 744 (1) insofar as a partially neutralised aliphatic amino acid is used in place of a free amino-substituted aromatic carboxylic acid and the reaction is initiated at a temperature between 90° and 100°C instead of being carried out at 65° to 121°C. The use of an aliphatic amino acid was obvious in the light of the disclosure in US-A-2 988 535 (2) in which aliphatic amino acids and aromatic amino acids are mentioned as alternatives. In the absence of any demonstration of a surprising effect the Examining Division considered that the subject-matter of the application did not involve an inventive step.

III. An appeal was lodged against this decision on 26 October 1984 with payment of the appropriate fee. In a statement of grounds filed on 28 December 1984, the Appellant argued that it was not permissible to combine documents (1) and (2), since the teaching of document (2) which relates to the preparation of fully cured polyepoxides has nothing to do with the problem of preparing water thinnable thermosetting binders. It was not obvious to select from the multitude of prior art documents the particular combination chosen by the Examining Division with the benefit of hindsight. The technical problem underlying the application was to be seen in improving the process disclosed in document (1) for the preparation of water thinnable thermosetting binders.

In a reply to a communication from the Board the Appellant argued that the technical problem did not lay in the preparation of products similar to the ones disclosed in the prior art but in finding a process that is advantageous over the one disclosed in document (1), the desired advantages being shorter reaction times "to be effected at lower reactivity". In the Appellant's view the proposed solution would be obvious only if it were known from the cited prior art that aliphatic amino acids react faster than amino-substituted aromatic carboxylic acids with epoxy groups. The disclosure in the Handbook of Epoxy Resins, H. Lee and K. Nesillo, pages 5-32 to 5-39 demonstrates that if a skilled person wished to produce water-soluble products similar to the prior art products he could choose from a large number of difunctional compounds. There is, however, no valid reason why he would select aliphatic amino acids. In a further communication from the Board it was pointed out that it would appear to be common general knowledge that the overall reaction rate of an amine with an epoxy resin is influenced by steric hindrance and the nucleophlic character of the amine. Therefore, it would appear that the skilled person would expect aliphatic amino acids to react faster than amino-substituted aromatic acids with epoxy resins.

IV. In the oral proceedings held on 1 December 1987 the Appellant submitted the results of two comparative examples demonstrating that it was essential to neutralise at least 50% of the acid groups of the aliphatic amino acid prior to the reaction with the polyglycidyl ether of a polyhydric phenol if the desired result of a short reaction time is to be achieved.

V. The Appellant requests that the decision under appeal be set aside and a patent be granted on the basis of Claims 1 to 8 and pages 1 to 13 of the description submitted during the oral proceedings.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is, therefore, admissible.

2. There are no formal objections to the present statement of claim since it is adequately supported by the original disclosure. The amended Claim 1 represents a combination of original Claims 1 and 6. Claims 2 to 8 correspond to original Claims 2 to 5 and 7 to 9 apart from the renumbering of Claims 7 to 9 and amendment of their dependencies as a consequence of the deletion of Claim 6.

3. The application-in-suit relates to a process for the preparation of thermosetting binders which are water-

soluble when neutralised with a base by reacting a polyglycidyl ether of a polyhydric phenol with an aliphatic amino acid in which at least 50% of the acid groups have been neutralised with an alkali metal hydroxide, a tertiary amine or a quaternary ammonium hydroxide in a ratio of from 0.7 to 1.2 amino hydrogen equivalent per epoxy equivalent. Document (1) discloses a process for the preparation of water-soluble compositions comprising reacting an amino-substituted aromatic carboxylic acid with a polyglycidyl ether of a polyhydric phenol and solubilising the resulting condensate by reacting the pendant carboxyl groups with an amine or ammonia (cf. Claim 1).

However, this prior art process was unsatisfactory insofar as the reaction times necessary to complete the reaction between the polyglycidyl ether of the polyhydric phenol and the amino-substituted aromatic carboxylic acid were excessively long. Thus, if the reaction is carried out at 80°C it is necessary to maintain the reaction mixture at this temperature for 12 to 15 hours to complete the reaction (cf. Example I). At a reaction temperature of 116 to 121°C a reaction time of five hours is required; an additional hour being required to raise the temperature of the reaction mixture from its initial temperature of 85°C to the above-mentioned reaction temperature (cf. Example IV).

4. In the light of this closest prior art the technical problem underlying the application-in-suit may be seen in providing a process for the preparation of water-soluble thermosetting binders from polyglycidyl ethers of polyhydric phenols in which the reaction times necessary to complete the reaction are shorter than those required in the above-mentioned prior art process.

According to the application-in-suit this technical problem is essentially solved by reacting the polyglycidyl ethers of the polyhydric phenols with aliphatic amino acids in which at least 50% of the acid groups have been neutralised with an alkali metal hydroxide, a tertiary amine or a quaternary ammonium hydroxide.

In view of the Examples where a reaction time of 15 minutes after initiation of the reaction by heating the reaction mixture to 100°C is disclosed, the Board is satisfied that the technical problem as defined above is plausibly solved.

5. After examination of the cited documents the Board has concluded that this technical teaching is not disclosed in any of them and the claimed subject-matter is, therefore, novel.

6. It still remains to be examined whether the requirement of inventive step is met by the claimed subject-matter.

6.1 In the light of the disclosure in document (1) a skilled person would reasonably conclude that aliphatic amino acids would condense with polyglycidyl ethers of polyhydric phenols in a similar manner to amino-substituted aromatic carboxylic acids, since it is clear from this document that the condensation takes place between the amino group and the epoxy group while the carboxylic acid group remains essentially unreacted (cf. column 2, lines 22 to 29).

6.2 The skilled person is also aware from his common general knowledge that the overall reaction rate of an amine with the epoxy group of an epoxy resin is positively influenced by an increase in the nucleophilic character of the amino group and from published pKa values that the amino group of an aliphatic amino acid, such as glycine or alanine, is more nucleophilic than that of an amino-substituted aromatic acid, such as 2-, 3- or 4-aminobenzoic acid. Therefore, in the light of these considerations a skilled person might have expected that the replacement of the amino-substituted aromatic carboxylic acids by aliphatic amino acids in the process disclosed in document (1) would solve the technical problem underlying the application-in-suit of reducing the reaction time of the prior art process.

However, as clearly demonstrated by the experimental evidence submitted by the Appellant during the oral proceedings the mere replacement of the known acids by the present ones did not solve this problem. Thus, in a repetition of the present Examples 1 and 4 using glycine and glutamic acid in the free acid form no reaction occurred at a reaction temperature of 90°C and a very slow reaction took place at 110°C (under pressure). Increasing the stirrer speed or removing all the water from the reaction mixture did not result in any increase in the rate of reaction. If, on the other hand, the glycine and glutamic acid are at least partially neutralised, the reaction proceeds to completion in 15 minutes at an initial reaction temperature of 100°C (cf. Examples 1 and 4).

In the light of the teaching in document (1) and his common general knowledge the skilled person could not have foreseen that the solution to the technical problem underlying the application-in-suit of reducing the reaction time of the process known from document (1) lay in using at least partially neutralised aliphatic amino acids in place of the free amino-substituted aromatic carboxylic acids in the process known from document (1).

6.3 Therefore, in the Board's judgement the subject-matter of the current Claim 1 involves an inventive step. Dependent Claims 2 to 8, which relate to preferred embodiments of Claim 1, derive their patentability from this claim.

Order

For these reasons, it is decided:

1. The decision under appeal is set aside.

2. The case is remitted to the Examining Division with the order to grant a patent on the basis of Claims 1 to 8 and pages 1 to 13 of the description submitted during oral proceedings.

The Registrar: The Chairman: