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D E C I S I O N
of 18 May 1994

Case Number: T 0064/92 - 3.3.3

Application Number: 86303101.9

Publication Number: 0200479

IPC: C08G 8/24

Language of the proceedings: EN

Title of invention:

Phenolic resins

Applicant:

INDSPEC CHEMICAL CORPORATION

Opponent:

-

Headword:

-

Relevant legal norms:

EPC Art. 56

Keyword:

"Inventive step - acknowledged after submission of appropriate comparative data"

Decisions cited:

-

Catchword:

-

Case Number: T 0064/92 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 18 May 1994

Appellant: INDSPEC CHEMICAL CORPORATION
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Decision under appeal: Decision of the Examining Division of the European Patent Office dated 22 July 1991 issued in writing on 22 August 1991 refusing European patent application No. 86 303 101.9 pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: C. Gérardin
Members: R. Lunzer
M. Aúz Castro

Summary of Facts and Submissions

- I. European patent application No. 86 303 101.9, Publication No. 200 479, was filed on 24 April 1986, having a priority date of 24 April 1985 derived from US Application No. 726 511.
- II. By its decision given on 22 July 1991, and issued in writing on 22 August 1991, the Examining Division refused the application, holding that the alleged invention was obvious in the light of documents:
- (1) US-A-3 328 354, and
 - (2) US-A-3 929 695.

That decision was based on a set of 18 claims, Claim 1 before the Examining Division, and also in accordance with the main request before the Board, being in the following form:

"A method for making a fusible phenol-resorcinol-formaldehyde resin solution, which resin solution comprises not more than 95 moles phenol and not less than 5 moles resorcinol per 100 moles of total phenol and resorcinol and from 0.55 to 0.75 moles of formaldehyde for each mole total of phenol and resorcinol, characterised in that said resin solution is prepared by the steps of:

(a) reacting said phenol and said formaldehyde at an initial pH in the range of from 8.5 to 9.2 in the presence of an alkali metal sulphite or bisulphite catalyst;

(b) then heating the reaction mixture to reflux until the mixture has a viscosity, when measured by

Gardner-Holdt method, in the range of from U to V Gardner-Holdt letters;

(c) introducing the resorcinol to the reaction mixture and continuing refluxing until the reaction mixture has a substantially constant viscosity in the range of from 30 to 100 Gardner seconds;

(d) then diluting the reaction mixture with a water-miscible solvent and adding an alkali metal carbonate in an amount by weight in the range of from 4 to 8 percent of the weight of the reaction mixture present at the completion of step (c) to obtain a resin solution having a solids content in the range of from 40 to 60 percent by weight; and

(e) then, if the pH of resin solution formed in step (d) is less than 7.5, adjusting the pH of said resin solution to a pH of at least 7.5."

Claims 2 to 12 are directed to preferred embodiments of the method according to Claim 1. Claims 13 to 18 concern phenol- resorcinol-formaldehyde resins obtained by a method either explicitly dependent on Claim 1, or incorporating essential features recited in Claim 1.

III. In its decision, the Examining Division observed that it was agreed by the Applicant that its alleged invention differed from the disclosure of document (1), the closest prior art, only in one respect; viz., that at the stage of diluting the reaction mixture with a solvent (step (d) in the above claim), 4-8% by weight of the reaction mixture, of an alkali metal carbonate, was added. As no comparison with the closest prior art was available, the relevant problem was regarded as being to increase the curing speed. However, as document (2) was concerned also with phenolic resin

adhesives to which resorcinol, formaldehyde and an alkali metal carbonate were added just before its application, and the carbonate had the effect of increasing curing speed, as could be seen from Table 7, columns 7 and 8, no inventive step could be recognised in adopting the same means, i.e. the addition of an alkali metal carbonate, with the objective of achieving the same effect, i.e. an increase in curing speed.

- IV. An appeal against that decision was filed on 16 October 1991, the appeal fee was paid on 23 October 1991, and the Statement of Grounds of Appeal was filed on 23 December 1991. In that Statement, and during oral proceedings held on 18 May 1994, the Appellant relied on further experimental data which had been filed with its grounds of appeal, and contended essentially that the Examining Division had been mistaken in looking at the problem solved by the alleged invention as being confined to securing faster curing.

It was normally to be expected that a reaction accelerator having the desired effect of reducing curing time would at the same time have the undesired effect of reducing the gelation time (i.e. pot life) below the desired level of at least 75 minutes, which was essential in adhesives intended to be used for wood lamination. It had been found, surprisingly, that the addition of alkali metal carbonates within the claimed range of 4 to 8% had three desired effects, viz. to improve curing times, while at the same time the desired relatively long gelation times were maintained, and in addition the strength of the bond was improved.

Although document (2) proposed the inclusion of alkali metal carbonates, that proposal was made in a wholly different context, and for a different purpose. Document (2) concerned resins formed by reacting phenols and formaldehyde to form a phenol/formaldehyde resin, to which resin, when used as an adhesive, a combination of formaldehyde, resorcinol and alkali metal carbonate was added. The systems in contemplation in document (2) and in the application in suit, although based on the same essential starting materials, were fundamentally different, as also were their objectives. Document (2) was concerned with thermosetting resins having a gelation time of some 11 minutes at 110°C. They were used in the manufacture of plywood, where curing by thermosetting was carried out at 135°C, not for room temperature wood lamination. The stated objective for adding the alkali metal carbonates was to improve the extendability of the resins (column 1, lines 3 to 4). Thus it afforded the skilled worker no suggestion of the possible advantages of including alkali metal carbonates in the system described in document (1), still less any suggestion to use the larger proportions of it, 4 to 8%, which had been an essential feature of the alleged invention from the outset.

- V. So far as concerned any improvement over the disclosure of document (1), the Appellant showed in an experimental report filed with its Statement of Grounds of Appeal that when comparing additions of 4% and 6% of potassium carbonate, with 5% of caustic soda, although the gelation times were comparable, the curing times were much shorter, with adequate bonding after some 4 hours, while a delamination test carried out after 24

hours showed that the strength of the bond in accordance with the alleged invention was very satisfactory, the percentage delamination with the carbonate being 0.7%, and 0.3%, contrasted with 20.5% with caustic soda. This reflected a significant increase in the strength of bond.

- VI. The Appellant requested that the decision under appeal be set aside and that a patent be granted on the basis of Claims 1 to 18 filed on 23 December 1991 and the description pages 1, 1/1 and 3/1 filed on 16 June 1990, pages 2 and 4 to 14 filed on 19 September 1989 and page 3 filed on 11 July 1991, as well as Figures 1 and 2 as originally filed.

Reasons for the Decision

1. The appeal is admissible.
2. *Admissibility of amendments*

As compared with the claims in the application as originally filed, the only amendments made are the deletion of the word "about" wherever used in Claims 1, 2, 4, 6, 7, 10 and 14. These deletions are clearly allowable for the purposes of Article 123(2) EPC.

3. *Novelty*

Having reviewed the cited documents, the Board is satisfied that none of them discloses a method for making a fusible phenol-resorcinol-formaldehyde resin solution having all the features defined in Claim 1.

Therefore the subject matter of Claim 1 is novel within the meaning of Article 54 EPC.

4. *The closest prior art*

It was agreed that document (1) is the closest prior art, and that, as stated in paragraph III. above, the claimed method differs from the known process only in that, at the stage of diluting the reaction mixture with a solvent (step (d) in the above claim), 4-8% by weight of the reaction mixture of an alkali metal carbonate is added.

5. *Problem*

5.1 The lack of any data comparing the performance of adhesives in accordance with document (1) with those in accordance with the alleged invention was filled by the experimental data mentioned in paragraph V above. These data show improvements in curing time and bonding strength, without loss of the desired relatively long gelation time.

5.2 The gelation times in these experiments are of the order of one quarter of the times given in Examples 1, 2, and 3 of the application in suit, notwithstanding the fact that the method used is said to be a repetition of Example 1, subject only to variation of the amounts of carbonate. However, whatever may be the source of this discrepancy, the important comparison is between the respective properties of compositions containing caustic soda, with compositions in accordance with the invention containing carbonate. In that respect there is consistency between Table I on

page 13 of the application as filed, showing the gelation times are comparable irrespective of which alkali is used, and the results shown in the experimental report, albeit that the times are much shorter.

- 5.3 Against the background of the experimental report, the Board identifies the relevant problem as being to shorten the curing time and to improve the bond strength, without loss in the desired relatively long gelation time, which is a necessary feature of adhesives to be used in lamination, as contrasted with those used in making plywood.

6. *Solution and its effectiveness*

As indicated above, the experimental report shows credibly that the use of an alkali metal carbonate, in place of the hydroxide previously used, has the desired effect of shortening curing time and improving bond strength, while leaving the gelation time substantially unaltered.

7. *Inventiveness*

- 7.1 As the claimed subject-matter differs from the disclosure of document (1) solely in the one respect, i.e. the use of an alkali metal carbonate, the issue to be determined here comes down to the simple question of whether a skilled person seeking to achieve improved curing speed and improved bond strength would have found in the above-mentioned documents any sufficient pointer towards using a carbonate.

7.2 Document (1), which emphasises the role played by the alkali metal sulphites in the adhesive properties of the phenolic resin, does not provide any incentive for such substitution. As indicated in this citation (column 3, lines 53 to 60), the lower limit of 0.04 mole of sulphite per mole of formaldehyde is essential not only for an efficient catalysis of the condensation reaction, but equally for the achievement of good adhesion properties, particularly with respect to delamination. Moreover, other alkalis being said to be unsuitable for the preparation of resins of that kind (column 3, lines 70 to 73), as illustrated in Examples I, VIII and XIII with caustic soda, the skilled person faced with the above-defined technical problem would have no incentive to look for alternative catalysts.

For these reasons, document (1) cannot lead the skilled person to the solution proposed in the application in suit.

7.3 Document (2) is concerned with a different problem, in a different area, in that it deals with improving the extendability of thermosetting resins to be used in the manufacture of plywood. More specifically, it is related to a method for preparing a rapidly curable and highly extendable phenolic resin adhesive which consists of adding a resorcinol, a formaldehyde and an alkali metal carbonate to a water-soluble thermosetting phenolic resin at room temperature just before its application. Although there is a reference at column 3, line 11 to accelerating the thermosetting speed, and at lines 62 to 65 to bonding times being markedly shorter, the highest proportion of sodium carbonate suggested is

3% in compositions of glues Nos. 6, 8, 17, 20 and 22. Furthermore, when comparing composition 15 with composition 16, and composition 18 with composition 19, it emerges that the addition of 1% of sodium carbonate has the effect of reducing the gelation time by some 30%, leading reasonably to the inference that additions of carbonate in the range of 4 to 8% might be expected to shorten gelation times still more.

Consequently, in the Board's view, the most that the skilled worker would derive from this disclosure is that the use of the alkali metal carbonate might accelerate the reaction at both stages, i.e. shorten the gelation time and curing time, but he would have had no reason to expect that with larger additions of carbonate, in a different system, gelation times would be substantially unaltered, whereas there would be a marked shortening in curing time and a significant increase in bond strength.

- 7.4 Accordingly, the Board is satisfied that it was not obvious to apply the suggestion of using the smaller proportions of alkali metal carbonates disclosed in document (2) to the system of document (1), while at the same time increasing the proportion of carbonate to 4 to 8% in the expectation of gaining the advantages achieved in accordance with the present invention.

8. *Conclusion*

Claim 1 being allowable, the same applies to dependent Claims 2 to 12, which are directed to preferred methods or processes according to Claim 1, Claim 13 which is a product by process claim, and to the product Claims 14

to 18, the inventiveness of which is supported by that of the main claim.

Order

For these reasons it is decided that:

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 18 filed on 23 December 1991 and the description pages 1, 1/1, and 3/1 filed on 16 June 1990, pages 2 and 4 to 14 filed on 19 September 1989, and page 3 filed on 11 July 1991, as well as Figures 1 and 2 as originally filed.

The Registrar:

The Chairman:

E. Görgmaier

C. Gérardin