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Bezeichnung der Erfindung: Carboxylated polyalkylene oxides and method for preparing the same
Title of invention:
Titre de l'invention :

Klassifikation / Classification / Classement :

ENTSCHEIDUNG / DECISION

vom / of / du 18 February 1988

Anmelder / Applicant / Demandeur : Union Carbide Corporation

Patentinhaber / Proprietor of the patent / Titulaire du brevet :

Einsprechender / Opponent / Opposant :

Stichwort / Headword / Référence :

EPÜ / EPC / CBE Article 56 EPC

Kennwort / Keyword / Mot clé : "Inventive step (denied) - Inadequate evidence"

Leitsatz / Headnote / Sommaire

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Case Number : T 159/84 - 3.3.2

D E C I S I O N
of the Technical Board of Appeal 3.3.2
of 18 February 1988

Appellant : Union Carbide Corporation
270 Park Avenue
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USA

Representative : Weinhold,Peter, Dr.
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Decision under appeal : Decision of Examining Division 011 of the European Patent Office dated 7 February 1984 refusing European patent application No. 79 104 670.9 pursuant to Article 97(1) EPC

Composition of the Board :

Chairman : P. Lançon
Members : G. Szabo
R. Schulte

Summary of Facts and Submissions

I. European patent application 79 104 670.9, filed on 23 November 1979 and published on 11 June 1980 with publication number 11 833, was refused by the decision of the Examining Division 011 of the European Patent Office dated 7 February 1984. The decision was based on Claims 1 to 6 received on 8 June 1982. Claims 1 and 6 were worded as follows:

"1. A method of preparing graft copolymers of acrylic acid and a polyalkylene oxide which comprises adding a solution of polyalkylene oxide and 3 to 15% by weight based on the total weight of poly(oxyalkylene) compound charged, of acrylic acid, and a solution of a catalytic amount of t-butyl perbenzoate, di-t-butyl peroxide or t-butyl hydroperoxide in polyalkylene oxide to an agitated bath of polyalkylene oxide, whereby all of said components are intimately admixed at a temperature of 100°C to 160°C and maintaining said temperature until a graft copolymer of acrylic acid on said polyalkylene oxide is obtained, said polyalkylene oxide having the formula:

$R'' \left|------(OC_nH_{2n}-)_zOR'\right|_a$

wherein R" is a hydrocarbon radical containing up to 10 carbons, free of aliphatic unsaturation, and having a valence of a, a is an integer having a value of 1 to 4; R' is a hydrogen or a monovalent hydrocarbon radical containing up to 6 carbons, free of aliphatic unsaturation, n has a value of 2 to 4 inclusive, and z is an integer having a value of 8 to 800.

6. Graft copolymers obtainable in the method of any of Claims 1-5."

II. The stated ground for the refusal was that the method claimed did not bring about any surprising results. The particular features selected for the purpose were all within the broad disclosure of US-A-3 418 354 (1) and their choice was obvious or a matter of simple trial and error. It was never demonstrated that the products according to the application were surprisingly better lubricants. No claims were allowed in view of lack of inventive step.

III. The Appellant lodged a Notice of Appeal against the decision on 9 April 1984 with the payment of the fee and submitted a Statement of Grounds on 18 June 1984. Thereafter, the Board issued several Communications, and the Appellant filed replies and the results of some tests on 6 October 1987 and 15 February 1988. An oral hearing was held on 18 February 1988.

IV. In the new set of claims filed by the Appellant on 8 August 1985, Claims 2, 3 and 5, were unchanged and Claims 1, 4 and 6, which were amended, have the following wording:

"1. A method of preparing graft copolymers of acrylic acid and a polyalkylene oxide which are substantially free of homopolymerized acrylic acid, characterized by adding a solution of ... (continued from line 3 of original Claim 1).

4. The method of Claim 1 wherein the polyalkylene oxide is polypropylene oxide started with a saturated aliphatic alcohol having up to 4 carbons.

6. Graft copolymers of acrylic acid and a polyalkylene oxide which are substantially free of homopolymerized acrylic acid and are obtainable by the method of any one of Claims 1 to 5."

V. The Appellant submitted in the Grounds for Appeal and at the oral hearing substantially the following arguments:

(a) The problem in relation to the closest prior art (1), Example 11, was to obtain a product with improved lubricating properties. The evidence showed that with the invention, a lower running temperature was obtained with a lower average force, and that this meant that the tool life would have been extended by about 10%.

(b) As regards the process of preparing the claimed products, the advantages manifested themselves primarily in the above superiority of the product, as well as in the consistent obtention of a hazeless material. It was not obvious to select the particular conditions for the process from the wide field of possibilities given in (1). Low temperatures were preferred and a specific order of adding the reactants was recommended. Although these possibilities, among others, were already indicated in the cited art, neither acrylic acid nor the separate addition of the reactant were preferred. The comparative tests in the specification showed that methacrylic acid would not have given the same advantageous result.

VI. The Appellant requests that the decision under appeal be set aside and the patent be granted on the basis of Claims 1-6 received on 8 August 1985 (main request) or alternatively on the basis of Claims 1 to 5 of the same set (auxiliary request).

Reasons for the Decision

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

2. No formal objections are raised against the amendments in the claims. The restriction of the claims to processes leading to products which are substantially free of homopolymerised acrylic acid is based on the description (page 4, lines 16 and 17). The obvious error in Claim 4 is now corrected.

3. The claimed subject-matter relates to the preparation of graft polymers of acrylic acid and polyoxyalkylene oxide. The closest prior art (1) describes a large group of polymers of such type. Acrylonitrile, styrene and ethyl acrylate are listed as preferred olefins for the purpose, since these produce clear and hazeless graft copolymers containing only small amounts of homopolymerised olefins (column 2, lines 53-56). Nevertheless, acrylic acid and methacrylic acid are also mentioned specifically, although no examples demonstrate the use of them. The products are stated to be useful, inter alia, as lubricants (column 7, lines 42-44).

4. The technical problem in respect to this particular closest state of the art could be seen either in the selection of graft polymers with a significant improvement of lubrication efficiency or, at least, in the provision of products which are equally hazeless and are therefore free of substantial homopolymerisation. The suggested solution of the problem is, according to the broadest Claim 6, a graft polymer obtainable according to the claimed methods, i.e. Claim 1. In essence, the preparation of the product involves the use of acrylic acid in an amount of 3 to 15% by weight, and of a specified catalyst and the separate addition of both in polyoxyalkylene (POA) solution into POA, at a temperature of 100 to 160°C.

5. In order to show improved properties, some results of tests with a turning lathe were presented on behalf of the Appellant in his letter from 6 October 1987. It was suggested the "lower the temperature and the lower the force, the better the lube". The figures showed a temperature of 193.3°C and a force of 512.5 lb with the material according to Example 5 of the application, whilst the temperature was 202.0°C and 515 lb with the material prepared according to Example 11 of (1) using acrylonitrile instead of acrylic acid. There were four different cut depths in each case and it took 30 to 60 seconds to run the lathe. No explanations could be given as to the basis of the averages and the spread of fluctuations in the circumstances. When the Board raised the question whether or not the figures could be influenced by lowering the force and allowing to run the lathe somewhat longer, i.e. up to 60 seconds, or by increasing the force and running it shorter, i.e. 30 seconds, to achieve the same depth, no explanations were offered. No conclusions could therefore be drawn as to the significance of the results.

6. The further submissions, arriving a few days before the oral hearing, did not strengthen the evidence either. The allegation that the result "is expected to cause an extension of tool life by about 10%" could not be substantiated at all. This was neither a statement of fact as having been observed with the concrete experiments, nor was it acceptable as common general knowledge in the art, on the basis of some known correlation between temperature and tool life. No authoritative corroborative statement or literature reference was submitted to impart credibility to the allegation which was therefore considered as filed late under Art. 114 (2) EPC and irrelevant in its content.

7. The Board already rejected some evidence filed earlier by the Appellant because of its inadequate character which did not enable the skilled person to repeat it and obtain the same results (cf. Communication 6.2.1986). The incompleteness of the presently available new results leaves the Board no option but to reject them as lacking conviction for the above reasons. The Appellant had, after all, five extensions of time to prepare his case and file his submissions and evidence. The consequence of all above discussions is that the problem cannot be the provision of an improved product, but it must merely be seen in the provision of an alternative product with secured hazelessness. To this problem, a solution has been credibly given in view of the examples of the application.

8. Although all essential features of the claims are separately disclosed in the cited art, the question of novelty was no issue in these proceedings. The particular combination of conditions specified in the present case was neither stated in (1) nor in other documents available to the Board, and thus novelty is to be recognised.

9. As regards the inventive step, the only relevant property established for the products of the invention is hazelessness, i.e. a low degree of homopolymerisation, if any. The claimed solution to the technical problem allows to achieve this with alternative embodiments.

As far as the quantity of the monomer is concerned it was known that reasonably low concentrations for the olefin were optimal, whilst too low values could lead to gel formation (cf. (1), column 6, lines 44-61). In view of the necessity of keeping control of the reaction, in particular when "an efficient peroxide, such as di-t-butyl peroxide is

used" (cf. column 6, lines 50-51), it was straightforward to opt for a low range, e.g. 3-15% total amount for the acrylic acid. It was, in any case, clear that the degree of homopolymerisation would be proportional to the amount employed. It is also relevant that the cited art (1) mentions that the preferred percentage is 10 to 100%, thus disclosing specifically the 10% value for the purpose (cf. Example 11 uses about 10%).

10. Acrylic acid is specifically mentioned in the cited art. Although this is not a preferred agent, the statement that the preferred ones produce clear and hazeless graft polymers cannot be construed in an exclusive sense, i.e. that no other kind can produce the same effect, in particular under conditions which are carefully chosen to avoid homopolymerisation. The fact that the appropriate limits are even lower for methylacrylate is not relevant in the circumstances. An exceptionally difficult situation with one specimen would not render normality in other cases unexpected. Thus, neither the choice nor the quantity employed was altogether unpredictable.

11. The same applies to the choice of temperature. Higher temperatures could have accelerated undesirable side reactions. In any case the citation specifically recommends 150°C as a lower value (cf. (1), column 5, line 53), and employs 160°C in some examples. The use of temperatures as high as 190°C for 55 minutes (in (1), Example 11) should not mean that a lower temperature with longer processing time would not have expected to provide similar results (cf. Example 1, 2,5 hours at 150°C). The range suggested in the present case is therefore not new in itself and has not shown to be decisively influencing the results.

12. The specific use of catalytic amounts of peroxides was also disclosed in (1). Di-t-butyl peroxide was repeatedly used

and there were warnings that large amounts thereof with low olefin concentrations could cause undesirable interactions and little of the desired graft copolymer (cf. column 1, lines 63-69). This and the instructions to keep "both the olefin and the catalyst concentrations" low (cf. column 6, lines 61-62) were sufficient for the skilled practitioner to adjust the conditions for the same catalyst to the required values. Thus, none of the specified conditions so far are alone or in combination to be considered as an inventive selection.

13. The only remaining feature to be considered is the manner of adding and mixing the reactants. Document (1) clearly envisages the possibility of adding the olefin and the catalyst separately to the POA medium (cf. column 2, lines 18-21), although the preferred way is to premix them beforehand (cf. also column 5, lines 30-38).

The Applicants found that the latter method involved the danger of premature reaction, thus leading to undesirable homopolymers, and followed therefore the second, alternative suggestion of the prior art. The addition of the reactants to a liquid component as a diluent first is a measure commonly used and recommended in reaction technologies. There can be no invention just in switching to the other, also recommended manner of carrying out a known process as soon as some difficulty is encountered, even if this is unexpected.

14. In view of the fact that the alternatives taken from the area already disclosed by the basic document (1) deliver no effect which is not already described in the document, nor do they do this unexpectedly in view of reasonable adjustments to achieve this, nor do they employ means undisclosed in the citation or under different conditions, the claimed process lacks inventive step. There was no

unexpected appreciable other property provided in the product which could have made that non-obvious as such and thereby render the process also patentable in consequence and therefore both Claims 1 and 6 are unallowable. The same applies to dependent subsidiary claims which impart no patentability to the case either.

Order

For these reasons, it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

F. Klein P. Lançon