

Veröffentlichung im Amtsblatt  
Publication in the Official Journal  
Publication au Journal Officiel

Ja/Nein  
Yes/No  
Oui/Non



Aktenzeichen / Case Number / N° du recours : T 55/88 - 3.3.1

Anmeldenummer / Filing No / N° de la demande : 83 306 034.6

Veröffentlichungs-Nr. / Publication No / N° de la publication : 0 106 627

Bezeichnung der Erfindung: Method for manufacture of aqueous suspension of  
Title of invention: solid organic peroxide  
Titre de l'invention :

Klassifikation / Classification / Classement : C07C 179/00

### ENTSCHEIDUNG / DECISION

vom / of / du 1 June 1989

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /  
Titulaire du brevet :

Nippon Oil & Fats Co. Ltd.

Einsprechender / Opponent / Opposant :

01 Kenobel AB  
02 Akzo N.V.

Stichwort / Headword / Référence :

EPO / EPC / CBE Arts. 56, 114(2) EPC

Schlagwort / Keyword / Mot clé :

"Inventive step (denied) - alternative method without proven advantage or overcoming of prejudice - comparative data disregarded as late and not relevant";

"Auxiliary requests not admitted as late and not clearly allowable".

Leitsatz / Headnote / Sommaire



Case Number : T 55/88 - 3.3.1

**D E C I S I O N**  
of the Technical Board of Appeal 3.3.1  
of 1 June 1989

**Appellant :**  
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Decision under appeal : Decision of Opposition Division of the European Patent Office dated 4 December 1987 revoking European patent No. 0 106 627 pursuant to Article 102(1) EPC.

Composition of the Board :

Chairman : F. Antony

Members : C. Gérardin  
G.D. Paterson

### Summary of Facts and Submissions

- I. European patent application No. 83 306 034.6 which had been filed on 5 October 1983, claiming Japanese priority of 12 October 1982, was granted as European patent No. 106 627 on 18 September 1985, with six claims, of which the only independent one, Claim 1, read as follows:

"A method for the manufacture of an aqueous suspension of a solid organic peroxide, characterised in that 5 to 70% by weight of a solid organic peroxide having a difference of at least 5°C between its melting point and its thermal decomposition temperature is mixed by agitation for a period of not exceeding 20 minutes with 0.1 to 10% by weight of a protective colloid, 0.1 to 20% by weight of a surface active agent and 10% to 80% by weight of water, at a temperature higher than the melting point and lower than the thermal decomposition temperature of said organic peroxide to form an aqueous emulsion of finely divided organic peroxide having an average diameter of not more than 30  $\mu\text{m}$ , said proportions being based on said aqueous emulsion, and subsequently cooling said aqueous emulsion."

- II. Notices of opposition were filed by

- (i) Kenobel AB on 26 May 1986 and by
- (ii) Akzo NV on 18 June 1986,

requesting complete revocation of the patent, for lack of inventive step over eight documents, including the following ones still referred to in the present appeal proceedings:

- (1) FR-A-2 474 510;
- (4) Derwent Abstract No. 76628X/41, relating to JP-A-76-96 783;
- (5) Research Disclosure No. 19 302 (May 1980);
- (6) US-A-3 849 148.

Later on, reference was also made to

- (9) Houben-Weyl, Methoden der Organischen Chemie, pages 102, 139 and 146.

The patent was defended with claims as granted, except that the peroxide percentage was limited to "10 to 70% by weight".

- III. In a decision orally announced on 4 November and posted on 4 December 1987 the Opposition Division revoked the patent in suit. The decision referred to by recognising an "essential difference" to the state of the art, implicitly accepted the existence of novelty, but negated an inventive step. According to the decision, it had been known to prepare peroxide suspensions by heating the peroxides to above their melting point and mixing them with a dispersing medium containing a surfactant and protective colloid. It was held to be obvious to obtain the desired particle size by choosing appropriate stirring and heating conditions, and stabilisation by means of protective colloids.
- IV. The Proprietor of the patent (Appellant) lodged an appeal on 27 January 1988, paying the required fee on the same date, and on 31 March 1988 submitted a Statement of Grounds, including certain amendment proposals concerning the claims. Further amendments to the claims were proposed in a written submission received on 1 December 1988.

The Appellant does not deny that, upon using the method of (1), it is possible to obtain suspensions similar to those resulting from the claimed method. He sees his invention as a new method for achieving storage-stable peroxide suspensions without the need for using colloid mills or similar special equipment subjecting the admixture to severe shear conditions. In his view, three mental operations were required for arriving at the invention:

- (i) the desirability of a protective colloid had to be recognised;
- (ii) an inclination to add the protective colloid only subsequently rather than together with the other ingredients had to be overcome;
- (iii) the perception had to be gained that simple stirring, without severe shear conditions, is sufficient for obtaining storage-stable suspensions from emulsions of molten peroxides.

This combination of mental operations involved an inventive step.

- V. The Respondents (Opponents) contest the existence of an inventive step. As it had been known, on the one hand, from (1) to obtain aqueous peroxide suspensions by applying high shear conditions to mixtures of peroxides, water, surfactants and protective colloid and, on the other hand, from (6) by forming emulsions of molten peroxide, water and surfactant and then cooling these emulsions, addition of protective colloid being mentioned in (6) as a possibility, it was obvious to combine the teachings of both documents. There was no inclination to add the protective colloid only subsequently, thus the above-mentioned mental operation (ii) could not contribute to an inventive step.

After the parties were summoned to oral proceedings, one of the Respondents (Kenobel) made reference to another document,

(10) US-A-3 988 261,

to show that it was known to use different types of apparatus for preparing emulsions or suspensions.

The Appellant submitted, on 30 April 1989, translations of the Japanese patent document forming the basis for (4), and of two pages from

(11) "Latest Processing and Modifying Techniques ..."  
(undisputed publication date: January 1981);

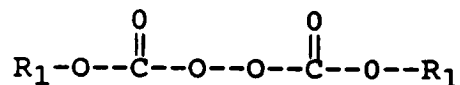
and by telefax of 24 May 1989 (confirmatory letter received the following day) comparative data intended to show an unexpected advantage of the claimed process over the prior art.

VI. At the oral proceedings of 1 June 1989, the Appellant, as his new main request, submitted a set of six claims, and he also indicated orally two auxiliary requests. Claims 1 and 2 of the main request read as follows (a typing error in Claim 1 corrected):

"1. A method for the production of an aqueous suspension comprising a solid organic peroxide, a surface active agent and water which comprises: mixing the suspension components and then raising the temperature with stirring to a temperature higher than the melting point and lower than the thermal decomposition temperature of the peroxide for a period of not exceeding 20 minutes, to form an aqueous emulsion, characterised in that 10 to 70% by

weight of the peroxide having a difference of at least 5°C, between its melting point and its thermal decomposition temperature is stirred with 0.1 to 20% by weight of a surface active agent, 10 to 80% by weight with water and simultaneously with 0.1 to 20% of a protective colloid, and subsequently cooling the so formed emulsion to provide a storage stable suspension having an average particle size of less than 30 μm.

2. A method as claimed in Claim 1, characterised in that the solid organic peroxide is a peroxydicarbonate represented by the general formula



wherein R<sub>1</sub> stands for an aliphatic hydrocarbon group having 3 to 18 carbon atoms."

The single claim of the first auxiliary request was said to be formed by combining Claims 1 and 2 of the main request together and with the enumeration of protective colloids given on page 4, lines 56 to 60, of EP-B-106 627.

The single claim of the second auxiliary request was directed to a combination of the single claim according to the first auxiliary request with the enumeration of agitators in the sentence of page 3, lines 38 to 39, and the stirring speed of about 600 rpm mentioned on page 7, line 6, of EP-B-106 627.

The Appellant asserted that, while the comparative data were occasioned by the late submission of (10), they were relevant in that they showed an unexpected advantage of the invention over the prior art represented by (1) and thus



supported the patentability of the claimed subject-matter. Accordingly he requested that the decision under appeal be set aside and the patent be maintained on the basis of amended claims in accordance with the main request or one of the auxiliary requests.

VII. The Respondents both maintained that the subject-matter of the patent in suit lacked an inventive step. The comparative data was late and should therefore be disregarded. Besides they did not represent a fair comparison to (1) and were therefore meaningless. The Respondents requested that the appeal be dismissed.

VIII. At the end of the oral proceedings the Chairman announced the decision of the Board that the appeal was dismissed.

#### Reasons for the Decision

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

#### Main Request

2. Although the claims in accordance with the main request were submitted at a very late stage, i.e. during oral proceedings, they are admitted for consideration having regard to the fact that Claim 1 corresponds largely to Claim 1 as submitted with the appeal grounds (with appropriate amendments requested on 1 December 1988); Claims 2 to 6 correspond to the granted version; and cancellation of Claims 7 and 8 as of 31 March 1988 has been effected in response to the Board's communication of 24 February 1989.

3. The formal admissibility of these claims has been considered and, except for an apparently unintended amendment in Claim 1 (the amount of protective colloid given as "0.1 to 20%" instead of "0.1% to 10%"), found in order. In view of the Board's decision to dismiss the appeal, this need not be discussed in detail.
4. Document (10) was filed late and is not considered relevant by the Board. It will therefore be disregarded under Article 114(2) EPC.
5. To the extent that the comparative data submitted on 24 May 1989 were in response to (10), they need not therefore be discussed. On the other hand, the Appellant also relies on such data as referring to the prior art in general and to (1) in particular, in order to support inventive step. To that extent, there is apparently, in the Board's view, no good reason why they could not have been submitted much earlier; accordingly they were not submitted in due time and would only be taken into consideration if the Board considered them relevant. No such relevance is recognised by the Board because, by the Appellant's own oral admission, the comparison made did not strictly apply the conditions of a particular piece of prior art, such as one of the Examples of (1) where a specific combination of mechanical dispersion and ultrasonic homogenisation is utilised; rather, the results of the invention were compared with those obtained using some, but not all parameters of (1). Hence the comparison is not relevant in support of inventive step and is therefore also disregarded under Article 114(2) EPC.
6. In relation to the results achieved, (1) is considered to be the closest prior art, it being not disputed that the process thereof gives rise to peroxide suspensions similar to those obtained according to the patent in suit, though

by a different technique. According to (1), the components of the desired suspension are mixed at room temperature (about 20°C) and finely dispersed by an apparatus exerting strong shear conditions ("Ultraturrax" at 10 000 rpm), followed by ultrasonic homogenisation (Example 1, page 11). As against this, in the absence of valid comparative data showing improved results, the problem underlying the patent in suit is to be seen in providing an alternative method for obtaining storage-stable aqueous peroxide suspensions, without application of strong shear forces to the sensitive peroxide particles. On the basis of the description of the patent in suit, it appears plausible that the claimed process does indeed solve the stated problem. This has not been disputed by the Respondents.

7. The claimed solution is novel, as none of the citations taken alone discloses all its features in combination. Novelty not being in dispute, no detailed reasons for this finding are required.
8. Existence of an inventive step remains to be investigated.
  - 8.1. Document (1), as set forth above, teaches a different technique for obtaining storage-stable aqueous peroxide suspensions, and does not in itself give any hint in the direction of the claimed process, i.e. of heating in conjunction with less vigorous agitation.
  - 8.2. While the method of (6) serves a very specific purpose (treating glass fibres to improve adhesion to polyolefins; see title) which does not necessarily call for storage-stability of the free radical initiator suspensions used ("free radical initiator" generally meaning "heat stable organic peroxide"; see column 2, lines 58 et sequ., and the specific compounds mentioned in Table I and the Table of Example II), peroxide suspensions are prepared by

thoroughly mixing the respective compound with a surfactant at a temperature above the former's melting point, then adding water still at a temperature above the said melting point, and finally cooling to ambient conditions (column 5, lines 34 to 46). This document thus teaches to apply to the art of peroxide suspensions the generally known technique of obtaining suspensions by first preparing emulsions and then cooling these, doing away with any reluctance which might previously have existed towards application of heat to even comparatively heat-stable peroxides.

- 8.3. The notional skilled person concerned with the problem underlying the patent in suit was looking for an alternative method for obtaining storage-stable aqueous peroxide suspensions without having to apply strong shear forces to the peroxide particles. In the Board's view, he would not in the least hesitate to try subjecting the starting mixture of (1) to sufficient heat for melting the peroxide involved, in combination with moderate agitation. He would have no prejudice against such heating, having read (6), and it would be most evident to him that the shear forces required to finely disperse a liquid -i.e. the molten peroxide -must be much smaller than those necessary to similarly disperse the peroxide in the solid state. The claimed method would hence not only be one of a number of techniques to be tried, but would be the first approach to be thought of, especially as protective colloids - an essential element of the storage-stable suspensions of (1) - are also mentioned as possible constituents in (6); see column 4, lines 44 to 47.
- 8.4. While, in the opinion of the Board, (1) and (6) would be sufficient to render the claimed alternate method obvious, prior art such as (4) and (5) would in any event serve to remove any doubts in the mind of the skilled person. If (4) teaches the preparation of an aqueous dispersion of

spherical peroxide particles by melting and thereafter cooling the peroxide in water in the presence of surfactants (the examples given for the latter being compounds acting at the same time as protective colloids), then clearly all that is required to obtain a finer, more storage-stable dispersion, i.e. a suspension, is to more finely divide the droplets of molten peroxide by moderate agitation.

- 8.5. In summary, in the Board's judgment the method of Claim 1 of the main request did not involve any inventive step.
- 8.6. No inventive contribution is seen in the selection of specific peroxides according to Claims 2 to 6, and moreover the main request must be refused as a whole once Claim 1 is not allowable.

#### Auxiliary Requests

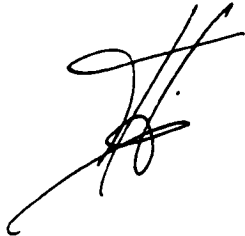
- 9. Both auxiliary requests were made at a very late stage, towards the end of oral proceedings concluding the appeal. In such a situation, the Board would only admit fresh claims to substantially different subject-matter if such subject-matter was clearly allowable (cf. T 153/85, "Alternative claims/Amoco", OJ EPO 1988, 1). This is not the case here, because it cannot be seen how a limitation to a certain class of peroxides in combination with an enumeration of protective colloids alone (first auxiliary request) or in further combination with an enumeration of certain types of agitators (second auxiliary request) could render the otherwise obvious subject-matter of the main request inventive; this is especially so as the Appellant has not advanced any arguments to that effect. Accordingly, the Board rejects the auxiliary requests.

Order

For these reasons it is decided:

The appeal is dismissed.

The Registrar



F.Klein

The Chairman



F.Antony

MP  
CG