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Bezeichnung der Erfindung: Suspension polymerisation of vinyl monomers

Title of invention:

Titre de l'invention :

Klassifikation / Classification / Classement : C08F 2/30

**ENTSCHEIDUNG / DECISION**

vom / of / du 30 November 1989

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /  
Titulaire du brevet :

The B.F. Goodrich Company

Einsprechender / Opponent / Opposant :

Hüls AG

Stichwort / Headword / Référence : Vinyl monomers/Goodrich

EPÜ / EPC / CBE Articles 56 and 100(b)

Schlagwort / Keyword / Mot clé : "Inventive step (confirmed)"  
"Sufficiency of disclosure (confirmed)"

**Leitsatz / Headnote / Sommaire**

Europäisches  
Patentamt

Beschwerdekammern

European Patent  
Office

Boards of Appeal

Office européen  
des brevets

Chambres de recours



Case Number : T 340/88 - 3.3.1

**D E C I S I O N**  
of the Technical Board of Appeal 3.3.1  
of 30 November 1989

**Appellant :**  
(Opponent)

Hüls AG  
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**Representative :**

**Respondent :**  
(Proprietor of the patent)

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**Decision under appeal :**

Decision of the Opposition Division of the European  
Patent Office dated 13 May 1988 rejecting  
the opposition filed against European patent  
No. 0 062 285 pursuant to Article 102(2) EPC.

**Composition of the Board :**

**Chairman :** K.J.A. Jahn  
**Members :** R.W. Andrews  
W. Moser

## Summary of Facts and Submissions

- I. The mention of the grant of European patent No. 0 062 285 in respect of European patent application No. 82 102 670.5, filed on 30 March 1982 and claiming priority of 1 April 1981 from a prior application filed in the United States of America, was announced on 12 February 1986 (cf. Bulletin 86/07) on the basis of fourteen claims. Claim 1 reads as follows:

"A process for the suspension polymerisation of vinyl monomer(s) in an aqueous medium in the presence of a dispersing agent(s) and a monomer-soluble polymerisation initiator with agitation and primary cooling in a polymerisation vessel having mounted thereon a reflux condenser which is open to the vessel throughout the entire polymerisation reaction, polymerising said monomer(s) by maintaining the reaction mixture at a temperature in the range of 40°C to 80°C, said process being characterised by continuously adding to the reaction mixture at a constant rate during the course of the polymerisation reaction from 0.003% to 0.5% by weight, based on the weight of said monomers, of a nonionic surface active agent having an HLB in the range of 9 to 16, whereby splashing of the reaction mixture and polymer build-up in said condenser are substantially eliminated."

- II. On 29 October 1988, a notice of opposition was filed requesting the revocation of the European patent on the grounds that its subject-matter lacked novelty and did not involve an inventive step. The opposition was supported, *inter alia*, by an English translation of JP-A-53 114 891 (3). After expiry of the time allowed for filing notice of opposition, the Opponent alleged that disclosure of the invention was insufficient.

III. By a decision dated 13 May 1988, the Opposition Division rejected the opposition on the basis that document (3) did not provide any indication that the solution to the problem of avoiding splashing, controlling charge expansion and eliminating polymer build-up in the reflux condenser during suspension polymerisation of vinyl monomers lay in continuously adding a nonionic surface active agent with an HLB value of between 9 and 16 at a constant rate to the polymerisation reaction mixture.

The Opposition Division disregarded the Opponent's allegation of insufficiency as regards the disclosure of the invention since it was not submitted in due time and was not supported by sufficient evidence.

IV. An appeal was lodged against this decision on 8 July 1988 with payment of the prescribed fee. A Statement of Grounds of appeal was filed on 27 July 1988. In his statement and during the oral proceedings held on 30 November 1989, the Appellant contended that the disclosure of the disputed patent was insufficient in the absence of any indication of how the HLB values referred to in Claim 1 are determined. Thus, it is not clear from the disputed patent whether the HLB values are those calculated from the theoretical composition of the surface active agent or experimentally determined values. However, even if it was assumed that the values correspond to analytically obtained results, the methods used to determine HLB values are not very accurate, as evidenced by the present practice of manufacturers to quote ranges of HLB values for their surface active agents in their technical literature.

Furthermore, the Appellant maintained that the skilled person would not be able to carry out the invention in view of the nomenclature used in the patent specification to describe the different types of surface active agents. For example, in certain instances the figure in brackets refers to the number of moles of ethylene oxide whereas, in other cases, it is intended that the figure in brackets should define the weight per cent of ethylene oxide. Finally, in this respect, the Appellant alleged that not all polyalkylene oxide block copolymers having the formula in Claim 6 have HLB values in the range 9 to 16 and also referred to the experimental results reported in his letter filed on 26 August 1987.

The Appellant also argued that subject-matter of the disputed patent did not involve an inventive step in the light of the teaching of document (3) combined with that of the brochure entitled "The new expanded Pluronic Grid" (document (4)).

- V. The Respondent argued that a combination of the disclosure of documents (3) and (4) show that only polyalkylene oxide block polymers having HLB values falling outside the claimed range are taught by document (3). In view of their position in the Pluronic Grid, these surface active agents can be seen to be excellent defoamers. In the Respondent's opinion, this demonstrates that the problem to be solved by document (3) was to prevent foaming. In contrast to this, the principle problem underlying the disputed patent was to eliminate or substantially reduce charge expansion by controlling the suspension viscosity. These two phenomena are different and the Respondent found that defoamers did not control charge expansion. Therefore, the combined teaching of documents (3) and (4) would not lead the skilled person to the present invention.

The Respondent also contended that at the priority date of the disputed patent, specific HLB values, rather than ranges of values, were assigned to surface active agents by the manufacturers. This can be seen from such standard works as "McCutcheon's Detergent & Emulsifiers, 1977 International Edition and the 1979 North American Edition". Furthermore, HLB is the only common parameter available to the skilled person for comparing surface active agents from different manufacturers. Even in the absence of any indication in the disputed patent of how the HLB values are to be determined and the differences in nomenclature, the Respondent considered that the disclosure of the patent in suit was sufficient to enable the skilled person to carry out the invention claimed therein.

- VI. The Appellant requested that the decision under appeal be set aside and the patent revoked. The Respondent requested that the appeal be dismissed.
- VII. At the conclusion of the oral proceedings, it was announced that the appeal was dismissed.

#### Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is, therefore, admissible.
2. The disputed patent relates to a method for eliminating or substantially reducing charge expansion, splashing and, to some extent, foaming, thereby preventing polymer build-up in the reflux condenser used as an external cooling means during the suspension polymerisation of vinyl monomers in the presence of dispersing agents and monomer-soluble polymerisation initiators.

- 2.1 It was found that when carrying out the suspension polymerisation of vinyl monomer, such as vinyl chloride, on a very large scale, the heat transfer area in the cooling jacket surrounding the polymerisation vessel was insufficient to cope with the heat generated during the polymerisation. In order to solve this problem, reflux condensers were mounted on the top of the polymerisation vessels so that the cooled condensate from the condensers could help to cool the polymerisation mixture. However, when using monomer reflux cooling, the volume of the polymerisation charge increased due to vapour trapped by the tendency of the polymer being formed to be carried to top of the charge by rising vapour bubbles. In addition, due to a change in the interaction between the polymer particles in the suspension, its viscosity increased at a later stage of the reaction causing an increase in the volume of the charge. When the vapour bubbles broke through the surface of the highly viscous expanded charge, splashing resulted which caused the reaction mixture to be carried up into the condenser where undesirable polymer build-up occurred. A solution to this problem would be to reduce the size of the charge so as to provide more space between the surface of the reaction mixture and the entrance to the condenser. However, this has the disadvantage that the productive capacity of the polymerisation vessel is reduced.
- 2.2 It was agreed by the parties that it was known to spray water into the reflux condenser in an attempt to solve this problem. However, this method was not considered to provide an entirely satisfactory solution.
- 2.3 In the light of this prior art, the technical problem underlying the patent in suit may be seen in providing an improved process for the suspension polymerisation of

vinyl monomers in the presence of dispersing agents and monomer-soluble polymerisation initiators in which polymer build-up in the reflux condenser attached to the polymerisation vessel, which is mainly the result of splashing of the reaction mixture due to its expansion during the course of the reaction and, to some extent, by foaming, is substantially eliminated.

- 2.4 According to the disputed patent, this technical problem is essentially solved by adding to the reaction mixture from 0.003 to 0.5% by weight, based on the weight of monomers, of a nonionic surface active agent having a hydrophile-lipophile balance (HLB) in the range of 9 to 16. According to Claim 1, the nonionic surface active agent is continuously added at a constant rate during the course of the polymerisation reaction. Although granted Claim 12 is rendered appendant to Claim 1, this claim is to be construed as an independent claim, since it claims an alternative solution to the above-mentioned technical problem, whereby the nonionic surface active agent having an HLB in the range of 9 to 16 is intermittently added to the reaction mixture.

In view of the Example in which the charge expansion was reduced from 5 cm to 0.5 cm by the addition of a nonionic surface active agent having an HLB of 11, the Board is satisfied that the above-defined technical problem is plausibly solved.

In the Board's judgement, the experimental results in the Respondent's letter filed on 26 August 1987 do not cast doubts on the above finding, since no details are provided regarding the exact experimental procedure and only information concerning the formation of foam and the flotation of the polymer particles is given. No reference is made to charge expansion, the prevention of which is



the most severe problem to be solved by the disputed patent.

3. After examination of the cited prior art, the Board has reached the conclusion that the subject-matter of the patent in suit is novel. Since novelty is no longer in dispute, it is not necessary to consider this matter in detail.
4. It still remains to be examined whether the requirement of inventive step is met by the claimed subject-matter.
  - 4.1 Document (3) discloses a process for the suspension polymerisation of vinyl chloride or mixtures of vinyl chloride with other copolymerisable monomers in an aqueous medium in which a copolymer of propylene oxide and ethylene oxide is present in the reaction mixture (cf. Claim 1 on page 1 of the English translation). According to the second complete paragraph on page 5 of this document, the purpose of adding these copolymers is to control the foaming of the reaction mixture brought about by the use of cellulose derivatives and/or polyvinyl alcohol as suspending agents. The control of foaming improves the fish eye characteristics of the resulting polyvinyl chloride.

Suitable copolymers of propylene oxide and ethylene oxide are prepared, for example, by copolymerising propylene oxide and ethylene oxide with propylene glycol or glycerol in the presence of a caustic alkali. Preferably, the copolymer contains from 10 to 20% by weight of ethylene oxide (cf. first complete paragraph on page 6). In addition, copolymers obtained using propylene glycol should have molecular weights in the range 2000 to 6000; in the case of those derived from glycerol the molecular

weight of the copolymer should be in the range 5000 to 9000 (lines 1 to 7 of the last paragraph on page 6).

According to lines 7 to 16 of the last paragraph on page 6 of document (3), in order to prevent foaming the amount of added copolymer should be at least 0.00005% by weight, based on the weight of vinyl chloride. However, to avoid a deterioration in the quality of the polyvinyl chloride due to the poor compatibility of the copolymer with vinyl chloride, the amount of added copolymer should be no greater than 0.02% by weight based on the weight of vinyl chloride.

Finally, according to the second paragraph on page 7, the copolymer may be present from the beginning of the polymerisation reaction or may be added successively at any rate during the course of the reaction, provided the concentration of the copolymer is within the range of 0.00005 to 0.02% by weight, based on the weight of vinyl chloride.

- 4.2 From his reading of document (3), the skilled person would conclude that the only problem addressed and solved by this document is the prevention of foaming during the suspension polymerisation of vinyl monomers. This view is supported by the disclosure of this document relating to the added copolymer. This document teaches generally the use of all copolymers which can be produced by known processes for copolymerising ethylene oxide and propylene oxide with propylene glycol or glycerol. Such copolymers would include both random and block ones. However, if the skilled person were to assume that the copolymers are block ones derived from propylene glycol and took into consideration the specified amounts of ethylene oxide and molecular weights, he could deduce from document (4) that the nonionic surface active agents of the Pluronic type

covered by this teaching are those falling within that area of the Pluronic grid encompassed by the points L101, L61, L62 and L92 where the most effective defoamers are to be found. This area is not covered by the claims of the disputed patent.

- 4.3 Furthermore, the skilled person would realise that foaming and charge expansion are two different phenomena. The former is dependent on the properties of the air/liquid interface and foams are destabilised by reducing either the surface elasticity or viscosity of the foam bubbles. Charge expansion is related to the wettability of the polymer particles and the interaction between them, thus resulting in a lava-like condition of the mass.

Therefore, document (3), which is wholly silent with regard to charge expansion during suspension polymerisation of vinyl monomers, would not provide the skilled person with any indication that the solution to the problem of eliminating it lay in using certain surface active agents of a specific type having HLB values falling within a definite range selected from the vast number of available surfactants (cf. McCutcheon's Detergents & Emulsifiers, 1977 International Edition, pages 1 to 199).

- 4.4 Document (4) describes the properties of Pluronic polyols, which are block polymers prepared by adding propylene oxide to the two hydroxy groups of a propylene glycol nucleus and adding ethylene oxide to both ends of the resulting hydrophobe base. The grid, which is formed by plotting molecular weight ranges of the hydrophobe against the per cent of the hydrophile in the final molecule, aids the skilled person in his search for the Pluronic or combination of Pluronics with the best balance of properties for the intended purpose. However, even combined with the disclosure of document (3), this

brochure would not lead the skilled person to the finding that the solution to the technical problem of eliminating charge expansion lay in using nonionic surface active agents having HLB values in the specified range.

- 4.5 Thus, in the Board's judgement, the proposed solution to the technical problem underlying the patent in suit is not obvious. Therefore, the subject-matter of Claim 1 is patentable.
- 4.6 Dependent Claims 2 to 11 and 14, which relate to preferred embodiments of the process according to Claim 1, derive their patentability from this claim.
- 4.7 For the above reasons, Claims 12 and 13, which claim the intermittent addition of the same nonionic surface active agent during the course of the polymerisation reaction, are also patentable.
5. After the above finding, it remains to be decided whether the belatedly submitted ground of opposition that the invention does not meet the requirements of Article 100(b) EPC should be considered by the Board in accordance with Article 114 EPC. In view of the Appellant's strong reliance on this point during the appeal stage, particularly during the oral proceedings, the Board considers it appropriate to take up this issue.
- 5.1 Although it is true that the disputed patent does not contain any information regarding the determination of the HLB value of the nonionic surface active agent, in the Board's opinion this would not prevent the skilled person from being able to carry out the claimed invention. Since it is well-known in the detergent field that the theoretical composition method for determining HLB may lead to considerable error, the skilled person would

reasonably assume that the claimed process should be carried out using nonionic surface active agents, the experimentally determined HLB values of which are in the range 9 to 16. This assumption is supported by the Example of the disputed patent in which Pluronic L63 is used as the surface active agent. According to Griffin's equation, the calculated HLB for a nonionic surface active agent containing 30% by weight of polyoxyethylene in the final molecule, in which the molecular weight of the polyoxypropylene hydrophobe base is 1750 (cf. document (4)), is 6, which is not in agreement with the value 11 cited in the disputed patent (cf. page 6, line 3). However, if the skilled person refers to standard works such as page 220 of McCutcheon's Detergents & Emulsifiers, 1979, North American Edition or page 74 of Handbuch der Kosmetika und Riechstoffe, Volume III, by Hugo Janistyn, he would find that the quoted HLB value of Pluronic L63 is 11.

- 5.2 With regard to the fact that for surface active agents of the generic classes (1) and (2) the figures in brackets are intended to indicate the number of moles of a particular component in the surface active agents (cf. Claim 7 and page 3, lines 62 to 65), whereas for those of generic class (3), it is intended that the figures in brackets should represent the per cent by weight of the component (cf. Claims 8 and 11), the Board is satisfied that this difference in nomenclature would not render it impossible for the skilled person to carry out the claimed invention. Thus, the nomenclature adopted for the generic classes (1) and (2) is standard and the skilled person would be in a position to select surface active agents of these classes, including those specifically mentioned, having HLB values in the range 9 to 16. It would be clear to the skilled person from the use of Pluronic L63 in the Example that the figure 30 in Claims 8 and 11 refers to

the per cent by weight of polyoxyethylene in the total molecule (cf. document (4)).

- 5.3 The fact that certain Pluronic surfactants falling within the scope of the formula in Claim 6 may have HLB values falling outside the range of 9 to 16 would not influence the skilled person's ability to carry out the claimed process, since the disputed patent clearly teaches that, to be suitable, the polyalkylene oxide block copolymer surface active agent must have an HLB value within the specified range, irrespective of the values of X, Y and Z in the formula in Claim 6.
- 5.4 Although at the priority date of the disputed patent it was recognised that the experimental method used to determine the HLB of a surface active agent was not very accurate, nevertheless, HLB was the parameter universally used to describe surface active agents, and specific values were assigned in the literature in this field to surfactants. Therefore, at this date the skilled person would have no difficulty in selecting nonionic surface active agents having HLB values in the range 9 to 16. The fact that it is present practice to quote a range for the HLB of a surface active agent does not alter the situation, since the skilled worker would select a surface active agent with an HLB range which overlaps to a great extent with the range specified in Claim 1; for example, a nonionic surface active agent having an HLB of 7 to 12 or 12 to 18 (cf. page 25 of the brochure relating to Pluronic & Tetcoric Block Copolymer Surfactants published in 1987 by the BASF Corporation).
- 5.5 In the light of the above, the Board considers that the patent in suit discloses the invention in a manner sufficiently clear and complete for it to be carried out by the skilled person.

**Order**

**For these reasons, it is decided that:**

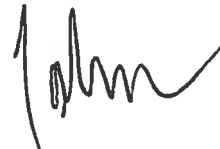
The appeal is dismissed.

**The Registrar:**



M. Beer

**The Chairman:**



K. Jahn