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Bezeichnung der Erfindung: Process for continuous controlled alcoholysis of  
Title of invention: polyvinyl acetate

Titre de l'invention :

Klassifikation / Classification / Classement : C08F 8/12

### ENTSCHEIDUNG / DECISION

vom / of / du 22 September 1989

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /  
Titulaire du brevet :

E.I. DU PONT DE NEMOURS AND COMPANY

Einsprechender / Opponent / Opposant :

HOECHST AG

Stichwort / Headword / Référence :

EPÜ / EPC / CBE Article 54(3)

Schlagwort / Keyword / Mot clé :

"Novelty (confirmed)"

Leitsatz / Headnote / Sommaire

Europäisches  
Patentamt

Beschwerdekammern

European Patent  
Office

Boards of Appeal

Office européen  
des brevets

Chambres de recours



Case Number : T 268/88 - 3.3.1

D E C I S I O N  
of the Technical Board of Appeal  
of 22 September 1989

Appellant :  
(Opponent)

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Decision under appeal :

Decision of the Opposition Division of the European  
Patent Office dated 19 April 1988 rejecting  
the opposition filed against European patent  
No. 0 054 716 pursuant to Article 102(2) EPC.

Composition of the Board :

Chairman : R. Andrews  
Members : C. Gérardin  
          W. Moser

## Summary of Facts and Submissions

- I. The mention of the grant of the patent No. 54 716 in respect of European patent application No. 81 109 224.6, filed on 29 October 1981 and claiming priority of 12 December 1980 from an earlier application in the United States of America, was published on 26 March 1986 (cf. Bulletin 86/13) on the basis of 13 claims.

Claim 1 reads as follows:

"In a continuous process for the partial alcoholysis of polyvinyl acetate homopolymer or copolymer which process comprises (1) rapidly premixing a methanol solution of polyvinyl acetate homopolymer or copolymer with a methanol solution of a basic catalyst in a mixing zone to form a uniform mixture of said solutions, (2) passing said mixture from the mixing zone into a reaction zone, (3) passing said mixture through said reaction zone while maintaining the temperature and residence time in said reaction zone such that the desired degree of alcoholysis is obtained, (4) continuously removing the resultant products of reaction from the reaction zone and (5) rapidly and uniformly neutralizing the basic catalyst in the resultant solid product by treating said product with an acidified liquid which is an inert non-solvent for the solid product, the improvement which comprises producing a partially alcoholized polyvinyl alcohol having a maximum hot water insoluble fraction of about 1.1% by weight by premixing catalyst and polymer solutions in a static mixer combined with using an intermeshing, self-wiping, twin-rotor mixer, or mixing extruder to provide the reaction zone, and by adjusting the feed temperature, the shear rate in the premixer, and the catalyst concentration in the catalyst feed solution such that the ratio

$$\frac{kC_i (10^2)}{\dot{\gamma}_p}$$

is from 0.5 to 1.4

where

$k$  is the alcoholysis reaction rate constant in  $\text{sec}^{-1}$

$C_i$  is the catalyst feed solution concentration in weight %

$\dot{\gamma}_p$  is the shear rate in the premixer in  $\text{sec}^{-1}$ .

- II. On 23 December 1986 the Appellant (Opponent) filed a notice of opposition requesting the revocation of the whole patent on the ground that the subject-matter of the patent in suit was not novel within the meaning of Article 54(3) EPC with regard to the teaching of EP-44 027 (document (1)).
- III. By a decision of 19 April 1988 the Opposition Division rejected the opposition on the ground that the requirement of novelty within the meaning of Article 54(3) EPC was met. More specifically, it was stated in this decision that, in contrast to the one-step process disclosed in document (1) which involved the use of an extruder-reactor, the process according to the patent in suit was a two-step process wherein mixing and reaction actually occurred in separate zones, whereby a narrow distribution of the degree of alcoholysis in the final product as well as both cold and hot water solubility were achieved.
- IV. The Appellant thereafter lodged a notice of appeal by a duly confirmed telefax on 21 June 1988 and simultaneously paid the prescribed fee. In his statement of grounds filed on 13 August 1988 as well as during the oral proceedings held on 22 September 1989, the Appellant put forward essentially the following arguments:

The difference between the process disclosed in document (1) and the claimed process regarding the number of steps could not be accepted, since the reactions involved were the same in both cases. In addition, the absence of any indication about water solubility in document (1) could not be used as an argument in favour of novelty, since saponified polyvinyl acetate polymers are well known to be soluble in cold water. Furthermore, the reference to a homogeneous mixture of the polymer solution and the catalyst solution in Claim 1 of document (1) served as evidence that homogeneity had already been achieved in the known process; this demonstrated that the static mixer could not provide the advantage claimed by the Respondent (Patentee) and that the relation between feed temperature, shear rate in the premixer and catalyst concentration in the catalyst feed solution was a superfluous characterisation likely to disguise lack of novelty.

- V. In his counter-statement filed on 23 December 1988 as well as during oral proceedings the Respondent mainly relied on the superior level of mixing achievable with a static mixer, as demonstrated in opposition procedure on 29 May 1987 by the inventor of the patent in suit. Since document (1) taught the use of a screw extruder not only for mixing purposes, but also as reaction zone, the issue of alleged lack of novelty could not reasonably be raised.
- VI. The Appellant requested that the decision under appeal be set aside and that the patent be revoked.

The Respondent requested that the appeal be dismissed.

At the end of the oral proceedings the decision 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 only question to examine is whether novelty within the meaning of Article 54(3) EPC can be acknowledged for the subject-matter of the patent in suit with regard to the teaching of document (1).
  - 2.1 This document describes a process for the preparation of polyvinyl alcohol by alcoholysis of a polyvinyl ester in presence of a basic catalyst and an alkanol as solvent, which comprises first dissolving a polyvinyl ester in 10 to 50% by weight of a lower alkanol in a reactor equipped with means for heating and cooling, then mixing the resulting solution at a temperature of 30 to 60°C with a solution of the catalyst in a lower alkanol, further warming the resulting homogeneous mixture to a temperature of 70 to 150°C, cooling the resulting polyvinyl alcohol to a temperature of 20 to 40°C, and finally recovering the polymer after the conventional neutralising, washing and drying steps (Claim 1).

The essential feature for the continuous operation of this process is the use of an extruder with one or two screws which acts both as mixing zone and reaction zone (column 2, line 64 to column 3, line 10; Examples 1 to 5). It may be true, as the Appellant put forward repeatedly, that the above sequence of operations is most comparable to the steps (1) to (5) mentioned in the preamble of Claim 1 of the patent in suit; however, whereas mixing and reaction are carried out according to the prior art disclosure in two different zones of one single apparatus,

the process claimed in the patent in suit requires the use of two different apparatus, each of them having one single specific function. Novelty can thus be acknowledged on the basis of a different concept regarding the apparatus features.

- 2.2 During oral proceedings, the Appellant emphasised that the disclosure of document (1) should not be regarded as being restricted to the use of extruders with one or two screws as actually mentioned, but more broadly as encompassing the use of any apparatus capable of ensuring the homogeneity of the mixture of the two solutions, such as a static mixer. In the Board's view, this argument is not acceptable for two reasons.

The first one, which results from the conclusion of the previous paragraph, is that the Respondent did not select a specific extruder or an alternative to extruders having both a mixing zone and a reaction zone, but chose instead a static mixer to carry out one single operation, namely the mixing operation; as indicated in the description of the patent in suit (page 4, line 18), it is important that relatively little reaction occur during this step, which confirms the specificity of the static mixer. The second reason is that the static mixer is not a mere alternative to conventional extruders as far as the level of mixing is concerned, but confers a much higher degree of homogeneity to the mixture, as the Respondent demonstrated in opposition procedure in the counter-statement filed on 29 May 1987.

In this reply, the inventor of the process claimed at present calculated the degree of mixing which can be obtained with an ISG (Interfacial Surface Generator) premixer from the number of layers generated by the successive subdivisions and recombinations undergone by

the fluid as it passes through the mixing elements (see as well the patent in suit, page 5, line 52 to page 6, line 11) and found that complete mixing on a molecular scale was actually achieved within a few seconds.

Likewise, the degree of mixing for laminar-flow systems in single screw extruders was estimated from the striation thickness parameter, as defined in the article "Mixing in Laminar-Flow Systems" by W.D. Mohr, R.L. Saxton and C.H. Jepson, Industrial and Engineering Chemistry, Vol. 49, November 1957, pages 1855 and 1856. The comparison of the two results shows that the use of an ISG premixer provides a degree of mixing higher by 7 orders of magnitude than the conventional extruders. In absence of any counter-evidence provided by the Appellant, who has the onus of proof, the Board relies on this empirical result to conclude that the static mixer is fundamentally different from the extruder disclosed in the prior art document.

- 2.3 For the same reason, the intermeshing, self-wiping twin-rotor mixer or mixing extruder required according to Claim 1 to provide the reaction zone cannot be equated to the single screw extruder exemplified in document (1).

As underlined in the patent in suit (page 4, lines 16 to 36 and page 5, lines 43 to 47), the time factor is an essential element of the process claimed by the Respondent. First, the static premixer enables not only the achieving of uniform mixing of the catalyst solution with the polyvinyl acetate-methanol solution as stated above, but also the obtaining of this result within very short residence times, in practice 0.04 to 0.6 minutes. As to the reaction itself, it has to be carried out for a carefully controlled length of time in a twin-rotor reactor that provides a narrow distribution of residence times (0.5 to 4 minutes) which itself contributes to keeping the vinyl alcohol ratio in the polymer within

narrow limits; as will be discussed hereinafter, this parameter is essential for the properties of the final product. Last, in order to avoid overreacted cold water insoluble fractions, quenching of the reaction throughout the reaction mass as soon as the desired degree of conversion has been reached, has to occur rapidly.

As the Respondent put forward, the necessity of having a narrow distribution of residence times in order to control the vinyl alcohol ratio in the end product and thereby the solubility thereof, is totally absent from the prior art teaching. The mere disclosure of alcoholysis taking place in the reaction zone of the extruder cannot therefore be interpreted as the implicit disclosure of the use of a twin-rotor reactor; the choice of this specific apparatus, which has to be considered in connection with the high degree of mixing achieved in the static mixer, i.e. with the quality of the mixture fed into the reaction zone, is thus a novel feature.

- 2.4 Additionally to the use of a static mixer as mixing zone and an intermeshing, self-wiping twin-rotor mixer or mixing extruder as reaction zone, the process according to the patent in suit requires the continuous control of the feed temperature, the shear rate in the premixer and the catalyst concentration in the catalyst feed solution such that the ratio defined in Claim 1 is from 0.5 to 1.4.

When used as protective colloids or stabilisers for polymer emulsions, it is essential that partially alcoholysed polyvinyl alcohols have good uniformity of composition and are soluble in water over a broad temperature range, i.e. exhibit simultaneously low cold water insoluble fractions as well as low hot water insoluble fractions (patent in suit, page 4, lines 23 to 25 and page 7, lines 55 to 61). This requires that the

distribution of degree of alcoholysis be over a narrow range from polymer to polymer within the composition. By contrast, the prior art process is not concerned with water solubility of polyvinyl alcohol at all, since the applications which are envisaged in this document call for good thermoplastic properties (column 3, lines 20 to 26).

For this reason, the relation between various process parameters in Claim 1 of the patent in suit cannot be regarded as a superfluous characterisation of the process likely to disguise lack of novelty. In the Board's view, on the contrary, this relation expresses the specific conditions which must exist continuously in order to achieve a uniform degree of alcoholysis. This teaching is not to be found in document (1) and cannot be inferred from it.

- 2.5 In conclusion, for the various reasons given above, document (1) cannot be regarded as an explicit or implicit anticipation of the subject-matter of the patent in suit which is consequently novel within the meaning of Article 54(3) EPC.
3. Claim 1 being allowable, the same applies to the dependent process Claims 2 to 13 which are based on the same combination of novel features and whose patentability is supported by that of the main claim.

Order

For these reasons, it is decided that:

The appeal is dismissed.

The Registrar:



M. Beer

The Chairman:



R. Andrews

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W. Moser 6.11.89

CG