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Bezeichnung der Erfindung: A process for the suspension polymerization of
Title of invention: tetrafluoroethylene
Titre de l'invention :

Klassifikation / Classification / Classement : C08F 14/26

ENTSCHEIDUNG / DECISION

vom / of / du 1 March 1990

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent /
Titulaire du brevet :

E.I. du Pont de Nemours and Company

Einsprechender / Opponent / Opposant :

Hoechst Aktiengesellschaft

Stichwort / Headword / Référence :

EPÜ / EPC / CBE Article 56

Schlagwort / Keyword / Mot clé :

"Inventive step after amendment of claim from
"process" to "use of compound in process"
(confirmed) - Functional relationship between
the use of a compound in a process and the
effect achieved - Claim limited to the context
where the technical effect is achieved"

Leitsatz / Headnote / Sommaire



Case Number : T 619/88 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 1 March 1990

Appellant : E.I. du Pont de Nemours and Company
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Respondent : HOECHST AKTIENGESELLSCHAFT, Frankfurt
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Representative :

Decision under appeal : Decision of the Opposition Division of the European
Patent Office dated 29 October 1987, posted on
11 October 1988, revoking European patent
No. 0 006 345 pursuant to Article 102(1) EPC.

Composition of the Board :

Chairman : F. Antony
Members : C. Gérardin
G. Paterson

Summary of Facts and Submissions

- I. The mention of the grant of the patent No. 6345 in respect of European patent application No. 79 301 124.8 filed on 12 June 1979 and claiming priority of 19 June 1978 of an earlier application in the United States, was published on 16 March 1983 on the basis of six claims.

Claim 1 reads as follows:

"A process for preparing a tetrafluoroethylene polymer which comprises the suspension polymerization of tetrafluoroethylene in the presence of an ionic radical initiator in an aqueous medium containing an aliphatic, substantially non-telogenic carboxylic acid to obtain a precipitated tetrafluoroethylene polymer, characterized in that said polymerization is carried out at a temperature of between 50°C and 100°C and at a pressure of between 10×10^5 and 50×10^5 Pa in an aqueous medium containing an aliphatic, substantially non-telogenic carboxylic acid of 1 to 6 carbon atoms and having a $-\log K$ between 1.5 and 10.0 in an amount between 25 and 2500 ppm, based on the weight of water present."

- II. On 14 December 1983, the Respondent (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 did not involve an inventive step with regard to the teaching of the following document:

(1) DE-A-959 060.

In a later submission the Respondent filed several additional documents in support of the same objection, especially

(2) US-A-2 393 767.

III. By a decision delivered orally on 29 October 1987 with written reasons posted on 11 October 1988, the Opposition Division revoked the patent on the ground that the requirement of inventive step was not met. More specifically, it was stated in that decision that the somewhat vague description of document (1) could only be interpreted as relating to a suspension polymerization process directed to the polymerization of a class of compounds embracing tetrafluoroethylene (referred to hereinafter as TFE). The control of the polymer particle size achieved there by addition of malonic acid according to Examples 1 and 2 was regarded as an effect similar to the reduction or even complete elimination of adhesions and clumps of resin particles along the walls of the reactor according to the patent in suit. The other features of the process claimed, especially the catalyst and the reaction conditions, such as pressure and temperature, were all considered to be usual in the art and their choice, in the absence of any surprising effect, did not involve an inventive step.

IV. The Appellant (Patentee) thereafter lodged a Notice of Appeal on 9 December 1988 and paid the prescribed fee at the same time. In the Statement of Grounds of Appeal filed by telefax on 21 February, confirmed in writing on 22 February 1989, the Appellant argued that the interpretation by the Opposition Division of the broad and vague description in document (1) concerning the monomer and the type of process used was not correct and that, on the contrary, document (1) had to be interpreted as

relating to a dispersion polymerization process tailored for the polymerization of chlorotrifluoroethylene (referred to hereinafter as CTFE).

- V. During oral proceedings on 1 March 1990, the issue of novelty was raised with regard to the teaching of Example VI of document (2). Thereafter, the Appellant filed the following amended Claim 1:

"The use of an aliphatic, substantially non-telogenic carboxylic acid of 1 to 6 carbon atoms and having a $-\log K$ between 1.5 and 10 to reduce the formation of adhesions which would otherwise form in a polymerizer equipped with a rotating internal agitator providing vigorous agitation during the suspension polymerization of tetrafluoroethylene to provide a precipitated tetrafluoroethylene polymer, the suspension polymerization being carried out in the presence of an ionic radical initiator in an aqueous medium containing between 25 and 2500 ppm of said non-telogenic carboxylic acid based on water present, and at a temperature of between 50 and 100°C and at a pressure of between 10×10^5 and 50×10^5 Pa."

According to the Appellant, this wording made it clear that the subject-matter of the patent in suit was directed to a process on industrial scale, unlike the prior art cited which was basically concerned with experiments on laboratory scale. For this reason, the features described in documents (1) and (2), although superficially similar to the solution required according to the process presently claimed, could not be transposed to the context of large scale production wherein the formation of adhesions along the walls of the reactor was caused by the vigorous internal agitation.

- VI. The arguments presented by the Respondent in the counter-statement of appeal filed on 5 August 1989 and during oral proceedings can be summarised as follows. The restrictive interpretation of document (1) by the Appellant could not be accepted since the teaching thereof concerned the polymerization of fluorinated vinyl monomers in general which encompassed TFE; as to the process, the necessity to carry out the polymerization reaction under agitation and the mention of the influence of acid treatment on the granular product in Example 2 were evidence that this prior art actually dealt with a suspension polymerization process. Similar conditions, especially the addition of an acid, were disclosed in Example VI of document (2) in connection with the suspension polymerization of TFE. The effect achieved in the prior art, i. e. reduction of the polymer particles size in document (1) and quantitative yield in document (2), could not be regarded in substance as different from the beneficial effect claimed by the Appellant.
- VII. The Appellant requested that the decision under appeal be set aside and the patent be maintained on the basis of the Claims 1 to 6 submitted during oral proceedings.

The Respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is, therefore, admissible.
2. The current version of the claims does not give rise to objections under Article 123 EPC.

In substance, the wording of Claim 1 differs from that of original Claim 1 in that it specifies that the polymerizer is equipped with a rotating internal agitator providing vigorous agitation. This feature is supported by the first paragraph of Example 1, read in conjunction with column 2, lines 14 to 16, of the patent in suit wherein the use of a polymerizer vigorously agitated at 600 rpm with an agitator is disclosed (column 4, lines 13 to 19), which in view of the blades mentioned in line 19 can only be interpreted as referring to an internal agitation. As to the formulation of Claim 1 as a use claim, this does not represent a true change of claim category, since it refers to the same use of the acid as defined in the original claims relating to a suspension polymerization process; for these reasons, the scope of Claim 1 does not extend beyond that of the original process claim.

The same applies to the dependent Claims 2 to 6 which have been redrafted as use claims accordingly.

3. The patent in suit concerns - although formulated as a use claim - a process for the suspension polymerization of TFE. Such a process is disclosed in document (2), which in the Board's view represents the closest prior art and comprises polymerizing TFE by contacting it, at a temperature of 0° to 200°C under a pressure of at least one atmosphere, with an inorganic peroxy compound and with water in a ratio of water to TFE within the range from 20:1 to 1:20 (Claim 2), whereby white granular polytetrafluoroethylene (hereinafter referred to as PTFE) suitable for molding operations is obtained in quantitative yield (page 1, column 2, lines 14/15 and 24 to 31). In order to avoid radical changes in pH during the course of the polymerization reaction, small amounts of materials which have a buffering action on the pH are usually added; since the polymerization reaction can be carried out

satisfactorily in either an acid or an alkaline medium, many types of buffering agents are operable (page 3, column 2, lines 4 to 14). For a pH below 7, acetic acid is mentioned as appropriate for this purpose and actually used in Example VI. According to the description, the reactor is charged with the various ingredients up to the desired pressure and is then agitated and heated to the desired temperature (page 1, column 1, line 55 to column 2, line 9); all the examples confirm that agitation is achieved by agitating the whole reactor. This is a distinguishing feature from the process presently claimed, wherein the presence of a rotating internal agitator providing vigorous agitation is required; novelty of the subject-matter of the patent in suit can thus be acknowledged at least on that basis.

4. As the Appellant put forward during oral proceedings, this difference in the way of achieving agitation of the reaction medium reflects in fact the difference in scale between the prior art and the patent in suit. The quantities of the various ingredients which are given in "parts", i.e. in relative amounts, in the examples of document (2) have to be appreciated in the light of the only absolute figure mentioned in the document, which is expressed in cm^3 (page 2, column 1, line 66); this means that the quantities of PTFE obtained vary between a few grams and at most 1 kg. Such polymer quantities clearly correspond to laboratory scale experiments. This ties in with the priority date of document (2) (1942), which shortly follows the discovery of PTFE.

In the light of this teaching the problem underlying the patent in suit can thus be seen in modifying the prior art process to make it compatible with the requirements of industrial scale production without impairing the granular properties and thereby the moulding ability of PTFE.

According to the patent in suit, this problem is solved by using a carboxylic acid specifically as defined in Claim 1 in combination with a rotating agitator providing a vigorous internal agitation of the reactor instead of agitating the whole reactor in presence of a buffer system.

In view of the examples of the patent in suit which illustrate the polymerization of TFE on a semi-industrial scale, the Board is satisfied that the above defined technical problem has been plausibly solved. This advantageous result has not been disputed by the Respondent.

5. It remains to be examined whether the claimed subject-matter involves an inventive step with regard to the prior art cited.
- 5.1 As stated above, the agitation of the reactor mentioned in document (2) is an external agitation of the whole reactor, whereby the reaction tube is rocked and its walls are washed by the reaction medium; for obvious mechanical reasons, the level of agitation which can be achieved by this method is only moderate. Whereas it is quite suitable for the type of reactors, such as the silver-lined pressure reactors mentioned in various examples, and more generally for the small-size equipment envisaged in document (2), it would be impractical in the case of large reactors; it is self-evident that no skilled person would consider agitating a large reactor. The solution of employing internal agitation, whose intensity may be adjusted to the high level claimed by the Appellant, could thus be regarded at first sight as obvious.

However, as the Appellant argued convincingly, the problem to solve was not just a problem of scale, thus size of equipment, for there are specific difficulties associated

with the use of an impeller with arms which do not appear in the case of external agitation of small reactors. These difficulties reside in the formation of adhesions, i.e. unwieldy large clumps of resin particles which form in the polymerization equipment, especially as annular build-up along the walls of the reactor, as the result of the polymer particles being flung against these walls, and may even clog the equipment; such adhesions will in any case reduce the yield of actually usable polymer. These problems do not occur in the small-scale process described in document (2) which, therefore, could not suggest a solution thereto.

Example VI of document (2), which describes the polymerization of TFE in the presence of acetic acid, has to be interpreted in the light of this conclusion. As mentioned above in point 3, this compound is added as part of a buffer system to prevent radical changes in pH during the course of the polymerization reaction (page 3, column 2, lines 4 to 25). Its action is not different from that of the other buffering agents cited which include as well strong acids, such as hydrochloric acid and sulfuric acid, and further various alkaline compounds, all compounds which are not envisaged in the patent in suit. From this list of compounds, it is evident that the use of acetic acid in Example VI cannot be the essential feature of the prior art process. The effect of these buffers can generally be appreciated from Example IV and VIII, which are comparative examples, wherein, in the absence of any such additive, very poor yields, respectively 16 and 1%, are obtained; thus, by controlling the pH, the buffering agent ensures a high degree of conversion of TFE into PTFE.

By contrast, the use of acids as defined in the patent in suit serves a different purpose. According to Example 1, 11.36 kg of TFE monomer give 12.25 kg of water-wet polymer and 1.18 kg of adhesions, thus 13.43 kg of wet polymer product altogether, when succinic acid is used; according to Comparative Example 1, in absence of succinic acid, one obtains 9.1 kg of water-wet polymer and 4.54 kg of adhered polymer, thus 13.64 kg of polymer product altogether, from the same amount of monomer. In both cases, thus, the total polymer yield is virtually the same, which shows that the acid has no effect on the degree of conversion; however, by lowering the polymer adhesions the acid increases the amount of usable polymer.

For these reasons, the high yields measured on experimental scale in document (2) when the polymerization reaction is conducted in presence of buffer systems containing acetic acid would not be an incentive to use the same acid in order to limit polymer adhesions when the process is performed under vigorous internal agitation on industrial scale.

- 5.2 Document (1), on which the Respondent relied more specifically and which was the basis for the decision of the Opposition Division, is a somewhat unreliable teaching both regarding the monomer and the type of polymerization process used. However, for the purpose of the present decision it is not necessary to discuss the scope of this document at length and to consider the possible influence of specific features, like the catalyst and the reaction conditions, on the polymerization reaction; for, even if one regards the definition of the monomer as encompassing TFE and the process as a true suspension polymerization process, this teaching cannot lead to or render obvious the subject-matter of the patent in suit.

Example 1 of document (1) describes the polymerization of CTFE in presence of a redox system; the reaction product is a non-water wettable product, whereas water-wettable, finely divided polymer particles are obtained when the reaction is carried out in the presence of malonic acid. In Example 2, which is the repetition of Example 1 for various amounts of malonic acid, it is specified that smaller quantities of acid result in coarser polymer particles (see in particular page 3, lines 2/3), which is interpreted by the Respondent as the possibility to reduce adhesions by merely adding carboxylic acid to the polymerization medium. This argument which is based on the analogy between the two phenomena - formation of polymer particles and formation of polymer adhesions - cannot be accepted for it overlooks that the formation of these adhesions requires the interaction between the polymer particles and the surfaces of the polymerization equipment, which is entirely different from the growing process of individual polymer particles in the reaction medium. As demonstrated by the Appellant during oral proceedings by means of samples, the size of polymer adhesions is incomparably larger than that of polymer particles. It is essential to bear in mind this difference when one analyzes the teaching of document (1); according to all the examples thereof, 100 grams of monomer are introduced into the reactor, which means that this disclosure, like that of document (2) above, should be regarded as an experimental study carried out on laboratory scale. For this reason, although a reactor with internal agitation is used (page 2, line 84), document (1) cannot suggest a solution to the problem of adhesions which occur on industrial scale. Furthermore, if the Respondent's assumption were correct, the elimination of adhesions would be accompanied by a reduction of the size of the polymer particles, thus a modification of the granular properties of the polymer; this does not occur in the process

according to the patent in suit, since the granular properties of PTFE are not affected by the presence of acid in the reaction medium (Example 1 and column 3, lines 58 to 61).

For these reasons, the control of the growing process of the polymer particles cannot be equated with the prevention of formation of adhesions, which makes the teaching of document (1) irrelevant for the solution of the above defined problem.

- 5.3 Unlike the prior art which is concerned with laboratory scale processes wherein the yield of the polymerization reaction can be improved (document (2)) and the size of the polymer particles can be reduced (document (1)) by addition of carboxylic acids, the patent in suit teaches the use of such acids to reduce the formation of polymer adhesions in the large scale polymerization equipment. For the reasons given above, this teaching cannot be derived from the prior art and is, therefore, inventive.

In the Board's judgement, the present formulation of Claim 1 brings to light the functional relationship which exists between the use in a suspension polymerization process of carboxylic acid as defined and the prevention of formation of polymer adhesions in a polymerizer equipped with a rotating internal agitator providing vigorous agitation.

6. Claim 1 being allowable, the same applies to the dependent Claims 2 to 6, which represent preferred embodiments of the subject-matter of Claim 1 and whose patentability 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 first instance with an order that the patent be maintained on the basis of Claims 1 to 6 submitted during oral proceedings.

The Registrar:

The Chairman:



M. Beer



F. Antony