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Aktenzeichen / Case Number / N° du recours : T119/82
Anmeldenummer / Filing No / N° de la demande : 79 301 547.0
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Bezeichnung der Erfindung: Process for the controlled gelation of polymeric
Title of invention: solutions and products thus obtained
Titre de l'invention :

ENTSCHEIDUNG / DECISION

vom / of / du 12 December 1983

Anmelder / Applicant / Demandeur : Exxon Research and Engineering Company

Stichwort / Headword / Référence : "Gelation/Exxon"

EPÜ / EPC / CBE

Articles 52(1) and 56
"Inventive step" - "Effects of processes" -
"Predictability of effect" - "Derivability
of invention from known effect"

Leitsatz / Headnote / Sommaire

- I. The effect of a process manifests itself in the result, i.e. in the product in chemical cases, together with all its internal characteristics and the consequences of its history of origin, e.g. quality, yield and economic value. It is well established that analogy processes are patentable insofar as they provide a novel and inventive product. This is because all the features of the analogy process can only be derived from an effect which is as yet unknown and unsuspected (problem invention). If, on the other hand, the effect is wholly or partially known, e.g. the product is old or is a novel modification of an old structural part, the invention, i.e. the process or the intermediate therefor, should not merely consist of features which are already necessarily and readily derivable from the known part of the effect in an obvious manner having regard to the state of the art (cf. also "Cyclopropane/Bayer, T65/83, OJ 8/1983, 327).
- II. Obviousness is not only at hand when the skilled man would have seen all the advantages of acting in a certain manner, but also when he could clearly see why he should not act in the suggested manner in view of

its predictable disadvantages or absence of improvement, provided he was indeed correct in his assessment of all the consequences.

III. Appellants who wish to rely on a prejudice which might have diverted the skilled man away from the alleged invention have the onus of demonstrating the existence of such prejudice.

Europäisches
Patentamt

Beschwerdekammern

European Patent
Office

Boards of Appeal

Office européen
des brevets

Chambres de recours



Case Number: T 119 / 82

DECISION
of the Technical Board of Appeal 3.3.1
of 12 December 1983

Appellant: Exxon Research and Engineering Company
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Decision under appeal: Decision of Examining Division 014 of the European Patent
Office dated 15 February 1982 refusing European patent
application No 79 301 547.0 pursuant to Article 97(1)
EPC

Composition of the Board:

Chairman: D. Cadman
Member: G. Szabo
Member: L. Gotti Porcinari

Summary of Facts and Submissions

- I. European patent application 79 301 547.0 filed on 1 August 1979 and published on 6 February 1980 with publication number 7815 claiming the priority of the prior application of 1 August 1978 (US-930 044), was refused by the decision of the Examining Division 014 of the European Patent Office dated 15 February 1982. The decision was based on claims 1 to 10. The main claim was worded as follows:

"A process for forming a polymeric solution or gel having a high viscosity which comprises:-

- (a) forming a solvent system of an organic liquid and a polar cosolvent, said organic liquid being an aromatic hydrocarbon, an aliphatic hydrocarbon, a chlorinated aliphatic hydrocarbon, a ketone, a cyclic aliphatic ether, an aliphatic ether, an aliphatic ester a paraffin oil or a mixture thereof, said polar cosolvent having a solubility parameter of at least 10.0 and being a water soluble alcohol, amine, di-or tri-functional alcohol, amide, phosphate, lactone, an acetamide or a mixture thereof, said polar cosolvent being less than about 15 wt % of said solvent system, the viscosity of said solvent system being less than about 1,000 cps;
- (b) dissolving a neutralized sulfonated polymer in said solvent system to form a solution, the concentration of said neutralized sulfonated polymer in said solution being 0.1 to 20 wt %, the viscosity of said

solution being less than 20,000 cps, the polymer having a backbone which has a solubility parameter of less than 10.5, the polymer having a level of crystallinity of less than 25% and the sulphonated polymer having been neutralized with cations of Groups IA, IB, IIA or IIB of the Periodic Table of Elements or lead, tin, antimony or ammonium or amine cations; and

(c) adding 5 to 500 volume % water based on the volume of organic liquid plus polar cosolvent to said solution, said water being immiscible with said solution, whereby said polar cosolvent transfers from said solution to said water causing the viscosity of said solution or suspension to increase from less than 20,000 cps to a higher viscosity."

II. The reason given for the refusal was that the subject-matter of claim 1 at least did not involve an inventive step. The cited publication, US-A-3 931 021, disclosed all the features of the claimed process for forming high viscosity polymeric solutions, except the use of water in the last step, and provided the same product as the applicants' process. Furthermore, the cited art revealed that any increase of the proportions of cosolvents in the mixture was associated with lower viscosities. Since it was also generally well known that excess of a polar cosolvent could be removed by extraction with water, the consequent increase in viscosity was not unexpected. Claim 10 related to the product of the process which lacked novelty and was likewise unallowable.

III. The applicants lodged an appeal against the decision of 15 February 1982 on 8 April 1982 with payment of the fee, and filed a statement setting out the grounds of appeal on 3 June 1982.

IV. In reply to objections raised by the Board, the appellant had submitted further arguments in good time and an oral hearing was then appointed for 12 October 1983. New sets of alternative amended claims were submitted before the hearing with the letter dated 20 September 1983, in which the first set represented a set originally filed with the applicaton, except for a limitation of the concentration range in step (b) to "0.5 to 5 wt. %" and for the deletion of claim 10. Otherwise this set was virtually identical with the claims cited in the decision of refusal save for minor amendments. In set B, the viscosity of the product was limited to a value greater than 50,000 cps and in set C the process also incorporated the steps of removing all solvents at elevated temperatures in order to deposit the polymer. Finally, in set D the same process was applied for well control by forming the gel in a well bore.

V. The grounds for the appeal and additional arguments submitted to the Board are essentially as follows:

(a) The cited US specification is only concerned with the problem of controlling the viscosity of polymeric solutions in the face of temperature changes. Any suggestion that it has anything to do with the increase of viscosity is inconsistent with its declared object. Gel is only mentioned in association with a non-polar solvent plus polar cosolvent in contrast to another statement which suggests that gels are formed by polymers capable of ionic cross-linking in non-polar organic liquids.

(b) The appellants also argue that conventional wisdom suggested that the addition of water would either precipitate a polymer or form a phase separated system. It is unexpected that with ionic polymers an emulsion is formed instead. It is also surprising that under certain conditions, i.e. at low polymer levels, the aqueous phase is gelled instead of the organic phase. Since the increase of alcohol content reduces viscosity it was not predictable that the addition of water would increase the same. To provide a viscous system in an organic solvent may take weeks.

(c) Furthermore the appellants challenge the assumption that their product is identical with that of the cited state of the art. The product of the invention consists of two phases whilst the state of the art represents a one phase system. Even if it were the same product, it would be wrong to deny patentability to an alternative process to obtain the same result since there is no explanation as to why the inventor would have contemplated the alternative route in the first place. In their opinion, whether or not the process is patentable is determined entirely by whether the steps of the process are obvious or not obvious. The nature of the product, e.g. its novelty, does not come into the matter; it is just the process itself which ought to be considered in relation to the prior art.

VI. The appellants requested that the decision under appeal be set aside and that the patent be granted. A refund of the appeal fee under Rule 67 EPC was also requested on grounds that the Examining Division had acted precipitately.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC, and is therefore admissible.
2. There can be no formal objection to the various sets of claims submitted with the letter dated 20 September 1983. All limiting features may derive support from the disclosure in the specification and claims as originally filed.
3. The problem with which the claimed process is concerned is to provide a polymeric solution or gel having high viscosity. The solution of the problem comprises essentially the dissolution of a neutralised sulphonated polymer in a mixture of a non-polar organic liquid and of a polar cosolvent, and the subsequent removal of most or all of the cosolvent by treatment with water. The method is illustrated by the examples in the specification, in particular with xylene or heptane as organic solvent and methanol, isopropanol and hexanol as cosolvents. Whilst with methanol the extraction with 8% water based on the volume of the organic liquid and cosolvent already raises the viscosity of the polymeric composition to over 50 000 cps, with isopropanol this is only achieved with 12% water. When hexanol is the cosolvent, its removal is much less efficient and with 14% water the viscosity is only increased to around 900 cps. Apart from the examples, a variety of polymers are individually recommended together with a list of specific organic liquids for each of them. The cosolvents are defined as having a "solubility parameter of 10.0" and being water miscible (page 9, line 33) or water soluble (page 10, line 12). It is only preferred that the cosolvents should "be soluble or miscible with the organic liquid"

although this requirement seems to be essential to achieve a transfer, i.e. extraction of the cosolvent with water.

4. The closest state of the art, US-A-3 931 021 (1), describes the formation of solutions of ionic polymers in a mixture of organic liquids and polar cosolvents, with specified viscosities. The same list of specific sulpho-nated polymers are individually recommended, together with a shopping list of organic liquids for each of them, as now presented in the application under appeal. The polar cosolvents are also generally characterised as having the same solubility parameter as in the present case, except that these must now be expressly "soluble or miscible" with the organic liquid. The recommended list ranges from methanol to much less polar cosolvents. In the working examples only hexanol, decanol and benzylalcohol are used. Nevertheless, a viscosity as high as 175 000 cps is achieved in a mixture of 1% hexanol in xylene.

5. Although the purpose of the examples is to show that the viscosities of the solutions resist temperature fluctuations more than those without cosolvent, the tabulated results also show that the viscosities are generally lower at higher cosolvent contents. In addition, the comparative tests show that the complete omission of cosolvent is still associated with one of the highest expected viscosities (e.g. 85 000 cps) in consequence of the effect of gradually reduced cosolvent content (Tables A, C, and D), if the halving of the polymer concentration is also taken into consideration (Table B appears to show a single anomalous exception). Notwithstanding the aim of the cited document, the Board is of

the view that the tabulated viscosities have relevant information content which were available for the skilled man aiming at high viscosities rather than the stability.

6. The above cited art confirms earlier experience with sulphonated polymers as thickening agents and in particular that "the addition of alcohols will reduce the viscosity of the thickened hydrocarbon" (cf. column 2, line 53 to column 3, line 4 referring to US-A-3 679 382 (2)). It was also known beforehand that organic liquids could be gelled with polymers having polar groups capable of forming "associating bonds" and that the presence of polar cosolvents was undesirable in this respect since it would reduce the effect (cf. column 2, lines 33 to 49 referring to US-A-3 666 430 (3)). These techniques were primarily concerned with the gelling of fuels, and advised against the use of cations other than ammonium only for reasons of deposition in the engine ((2), column 4, lines 36 to 44). Nevertheless they established a relevant background as to the formation of gels with ionic polymers in organic liquids. Although there is no obvious reason why the addition of minute amounts of water enhanced the thickening of compositions in one instance (cf. (2), column 4, lines 44 to 49), the observation is contrary to the arguments of the appellants that a precipitation of the polymer would have been expected in such circumstances.

7. As to the limits which the references to solubility or miscibility with organic liquids, on the one hand, and water, on the other, imply for cosolvents, it is relevant that the specification under consideration specifically mentions acetamide and 1,2-propane diol (page 10, lines 13 to 17). These are slightly soluble or soluble i.e. not totally miscible with benzene, a repeatedly mentioned organic liquid. The same are merely soluble

and miscible, respectively, with regard to water. The requirement for solubility in water must therefore be construed so as to include even slight solubility as is the case with hexanol. The term in this context can only mean that the cosolvent must be capable of being extracted to a large if not full extent with water to increase the viscosity of the remainder considerably. Although hexanol was only slightly removed with 14% water in the Example, the claims refer to the possible use of 500% water. The corresponding 35- fold increase in the degree of extraction of hexanol with such excess of water would provide substantially higher viscosities than those recorded after the use of small amounts of water. There is no reason therefore to exclude the slightly water soluble hexanol from the scope of the claimed process, neither was there any argument or submission for amendment from the appellants to imply anything to the contrary.

8. Since the examples of the application also demonstrate results with less than maximum viscosity by using smaller quantities of water for extraction, effective amounts of cosolvents must have been retained in the organic liquid after the single treatment to account for the differences. The resulting products should therefore fall within the general class of viscous three-component mixtures outlined in the cited art (1). Alternatively, if the claimed process were to lead substantially to viscous two-component systems without any effective cosolvent left in the organic phase, the results should fall within the area of or closely resemble the products to the earlier art, in which ionised polymers simply gell or thicken organic liquids, i.e. (2) and (3). It is also relevant that even (1) describes specifically at

least one highly viscous composition containing also hexanol, and also other such compositions in pure xylol (cf. above under 4 and 5).

9. The appellants have argued that the products obtained according to the claimed process are novel and not identical with those suggested in the state of the art, since there is also an aqueous phase present. It is evident, however, that the aqueous phase is inessential and useless, and should, even if entrapped in the gel, lessen the overall viscosity of the result. In larger amounts, it would even defeat the purpose of the exercise if left around the product and this is why the specification recommends its removal by conventional "liquid extraction" methods (page 4, line 2) which obviously mean appropriate separation techniques. Alternatively, the aqueous phase may be simply evaporated (page 2, line 36 to page 3 line 7, and claim 2). The temporary aggregation of a known article with functionally superfluous and unrelated other known components is representing a mere collocation of objects, which should not impart novelty to the same unless there are reasons to the contrary suggesting some further effect. An increased amount of an undesirable contaminant would not, for instance, render an old compound new. In the present context, the aqueous phase is a by-product of no technical relevance.

10. The product range of the process of the application shows a considerable overlap with the gelled liquids of the state of the art, and embraces specifically a few already known compositions. Even if the latter were to be removed by disclaimer to restore novelty, the remaining range would not necessarily represent a novel and

non-obvious selection in distinction to the general class of gelled two- or three component liquids. There was no suggestion that the viscous solutions or gels provided by the application would have unforeseen advantageous properties in this respect, and were not claimed as such. The patentability of the claimed process for preparing the same cannot, therefore, rely on the patentability of the product.

11. Contrary to the arguments submitted on behalf of the appellants which suggested that the nature of the product should not come into this matter at all, the Board takes the view that the character of the product, including its novelty and obviousness in the light of the state of the art, has a decisive role in the assessment of the inventive step for the process. The effect of a process manifests itself in the result, i.e. in the product in chemical cases, together with all its internal characteristics and the consequences of its particular history of origin, e.g. quality, yield and economic value. It is well established that analogy processes, i.e. which are themselves otherwise non-inventive, are patentable insofar as they provide a novel and inventive product. This is because all the features of the analogy process can only be derived from an effect which is as yet unknown and unsuspected (problem invention). If, on the other hand, the effect to be attained is wholly or partially known, e.g. the product is old or is a novel modification of an old structural part, the invention, i.e. the process or the intermediate therefor, should not merely consist of features which are already necessarily and readily derivable from the known part of the effect in an obvious manner having regard to the state of the art (cf. also "Cyclopropane/Bayer, T 65/83, OJ 8/1983, 327).

12. The relevant features of the product at hand were its components, i.e. the sulphonated polymer, the organic liquid, and a particular amount of the cosolvent or none of it, as well as the required viscosity value. Such known mixtures are obviously obtainable by admixing the components and by subsequently adding, if necessary, further amounts of the components in order to make up for any deficiency and to adjust the viscosity to the desired value. The definition of the viscous product with all its characteristics, therefore, implies its own solution in process terms to the skilled person in the art. As to the alternative processes to the same end, the question arises whether or not the removal of certain excess components for the purposes of adjustment, instead of the addition of others to make up for a deficiency, would have been also obvious in the circumstances. Such tasks directly involve separation techniques well known as basic unit operations in chemical engineering, and may include evaporation and extraction techniques. As for the readjustment of the cosolvent content of any three-component mixture by removing an excess, the skilled man would have readily recognised extractions with water as a feasible solution in view of the presence of substantially non-polar organic liquids. The result, unlike in many instances in chemistry, would have been readily predictable on the basis of available data on solubilities, distribution coefficients, and simple physico-chemical considerations.

13. The statement of the inventor that he was not conscious of the consequences of the addition of water cannot refute the presumption that the notional skilled man would have been aware of the outlined possibility in the absence of anything to the contrary. The suggestion that the cited article by Longworthy ("Ionic Polymers", edited by Holliday, 1975) discussed a plasticisation effect with water and indicated a considerable degree of hydration of the ions is of no consequence. This effect was, of course, observed in the absence of non-polar liquids which would have been expected to represent a very hostile environment for water. Whilst other polar cosolvents have both hydrophilic and hydrophobic components implying solubilities in both polar and non-polar solvents, water is only polar in character and cannot be easily envisaged to survive in non-polar environments as a solvating agent to any appreciable extent. The appellants were invited to submit evidence in this respect but they offered none which would have indicated that the product was, against expectation, hydrated in such media after all. Similar considerations would also explain the allegedly unexpected formation of emulsions with gels, where apart from physical entrapment of small amounts of dispersed water in highly viscous systems, the hydrophilic/hydrophobic character of the ionic polymer may also play a stabilising role.
14. The submission that rather a precipitation of polymer was expected than a viscous solution, is also unconvincing. The example referred to by the appellants was polystyrene which contains no hydrophilic sulphonate groups. Furthermore, such separation would have only been expected if the solvent could dissolve appreciable amounts of

water, which is not the case with the substantially non-polar liquids to be used. The additional argument that water itself could be gelled instead of the organic phase when the sulphonated polymer is used in low concentrations, is an allegation without any support. Appellants who wish to rely on prejudice which might have diverted the skilled man away from the invention have the onus of demonstrating the existence of such prejudice. A mere reference to an unpublished pending patent application, which is not available to the public and to the Board, cannot refute the impression that the submission is, on the face of it, without credibility. It implies that an amount of the sulphonated polymer above the suggested limit would have removed the cause of the unexpected gelling of the aqueous phase instead of increasing its quantity, if anything, in both phases. This is a totally unexpected proposition which cannot be entertained without very convincing evidence in support thereof. The content of this allegation and the manner of its presentation are contrary to established practice and, in particular, to the principles of handling evidence before any judicial tribunal.

15. The appellants submitted no evidence which would have either shown that a real prejudice must have existed against contemplating the use of water for adjusting the viscosity or that there is some unexpected advantage when the claimed process is used, in comparison with the direct mixing process. The new indirect approach, incorporating the additional step of extraction and, optionally, the removal of the aqueous phase would appear to be an additional burden or detour, which can only be offset by some yet unsuspected further effect. The

claimed process may itself take 24 hours to complete and there was no evidence in support of the allegation that the direct route would take much longer. The modification of the state of the art, i.e. the mixing technique in itself, with the extraction technique has apparently no unsuspected characteristics or advantages.

16. The argument that alternative routes should be considered to be the less obvious the odder, or perhaps even the more disadvantageous, they are, cannot be sustained. The rhetorical question why the skilled man should have contemplated such detour at all, would equally apply, if someone tried to patent the least attractive further analogy processes for the making of a known compound. Obviousness is not only at hand when the skilled man would have seen all the advantages of acting in a certain manner but also when he could clearly see why he should not act in the suggested manner in view of its predictable disadvantages or absence of improvement, provided he was indeed correct in his assessment of all the consequences

17. The product of the claimed process is known in the art and is specifically characterized by its components and the desired viscosity. The former features imply all necessary components for an alternative process to obtain the mixture and viscosity is known to be governed by the exact level of cosolvent content. As explained, the adjustment of that level by the removal of any excess thereof was already available in the state of the art through appropriate extraction methods with water.

The application of all the claimed measures for mixing and adjusting the composition were readily derivable from the state of the art and therefore obvious, and the decision of the Examination Division that the main claim (set A) lacks inventive step must be confirmed.

18. The features added in the auxiliary set of claims B and C represent nothing which could impart patentability to any one of the main claims. The limitation to 50 000 cps viscosity values in set B represents products which were equally predictable and obtainable in view of the highest viscosities disclosed for both three- and two-component systems in the closest state of the art (1). The limitation to coating a substrate in set C, which involves the evaporation of all solvents to deposit the dry content of the gel, is a technique clearly commonly known for all viscous solutions or gels in the paint industry. The position with regard to set D relating to the control of wells is, on the other hand, as yet impossible to assess since this may involve circumstances and interactions under conditions peculiar to themselves, and in the absence of search, demonstrating the state of the art with regard to the feasibility of gelling in such situations, the use of such known gels and the character of effect cannot be properly estimated.
19. Since the explanations submitted on behalf of the applicants during prosecution before the Examining Division had given insufficient basis to refute the serious presumption of obviousness, and the Examining Division could therefore see no grant even on the basis of amendments, the issuance of the refusal was justified (cf. also the decision of the Technical Board of Appeal,

(T 84/82 "Chloral Derivatives", 18.3.1982, to be reported). In any case, the refund of the appeal fee can only be entertained when there has been a substantial procedural violation, which is, in the opinion of the Board, inapplicable to the issues of the present appeal.

Order

It is decided that

1. The decision of the Examining Division 014 of the European Patent Office dated 15 February 1982, is set aside.
2. The appeal, insofar as it relates to claim sets A, B and C submitted with the applicants' letter of 20 September 1983, is rejected.
3. The application is remitted to the first instance for further substantive examination on the basis of claim set D relating to well control, also submitted on that date.
4. The request for reimbursement of the appeal fee is dismissed.

Registrar:

Chairman

J. K. K.

12/11/83

12/12/83

J. T. Cadman