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Datasheet for the decision
of 6 April 2017

Case Number: T 1239/13 - 3.3.03

Application Number: 02794800.9

Publication Number: 1432753

IPC: C08G77/26

Language of the proceedings: EN

Title of invention:
POLYSILOXANES AND THEIR PREPARATION

Patent Proprietor:
Dow Corning Corporation

Opponent:
Evonik Degussa GmbH

Headword:

Relevant legal provisions:
EPC Art. 56

Keyword:
Inventive step - (yes)

Decisions cited:

Catchword:

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Case Number: T 1239/13 - 3.3.03

DECISION
of Technical Board of Appeal 3.3.03
of 6 April 2017

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Decision under appeal: **Interlocutory decision of the Opposition Division of the European Patent Office posted on 18 March 2013 concerning maintenance of the European Patent No. 1432753 in amended form.**

Composition of the Board:

Chairman D. Semino
Members: M. C. Gordon
C. Brandt

Summary of Facts and Submissions

I. The appeal lies from the interlocutory decision of the opposition division posted on 18 March 2013 according to which it was held that European patent number 1 432 753 could be maintained in amended form on the basis of the main request as filed at the oral proceedings before the opposition division.

II. The patent was granted with a set of 20 claims, whereby claim 1 read as follows:

"A process for the preparation of an amino-functional polysiloxane comprising reacting an aminosilane (A) which contains an aminoalkyl group and at least one alkoxy group bonded to Si with a carboxylic acid (C) and a silanol-functional polysiloxane (B), the amount of carboxylic acid (C) being such that the molar ratio of carboxylic acid groups of (C) to amino groups of aminosilane (A) is less than 1:1 and the amount of silanol-functional polysiloxane (B) being such that the molar ratio of silanol groups of (B) to Si-bonded alkoxy groups of aminosilane (A) is greater than 1:1, whereby the aminosilane (A) is partially converted into its carboxylate salt which acts as a catalyst for the siloxane condensation polymerisation reaction between (A) and (B)".

III. A notice of opposition against the patent was filed invoking the grounds pursuant to Article 100(a) EPC (lack of novelty, lack of inventive step) and Article 100(b) EPC (insufficiency of disclosure).

The following documents were, *inter alia* relied upon by the opponent:

E1: US-A-4 496 705
E2: DE-OS-197 39 964
E3: EP-A-417 047
E4: US-A-5 621 060
E5: WO-A-99/06486.

IV. The decision of the opposition division was based on a set of claims 19 claims submitted in writing and further amended at the oral proceedings before the opposition division. Claim 1 of this set of claims was identical to claim 1 of the patent as granted.

According to the decision, novelty was acknowledged, which finding was not been challenged in the appeal proceedings. An inventive step was recognised starting from document E2 as the closest prior art. It was not obvious to replace the Lewis acid fluoride catalyst of E2 by a carboxylic acid catalyst as defined by the operative claim as a solution to the problem of providing a further process for the preparation of an amino-functional polysiloxane with a regular distribution of aminosilane-derived units throughout the molecule.

V. The opponent (appellant) lodged an appeal against the decision, maintaining objections pursuant to Article 56 EPC in respect principally of E2 as the closest prior art. A separate attack of lack of inventive step was formulated on the basis of E1 as the closest prior art.

Two further documents were submitted, namely:

E9: M. G. Voronkov and Yu.A. Yuzhelevskii: "The siloxane bond - Physical Properties and Chemical Transformations" (1978)
E10: US 4 446 283.

The objections were pursued in a second letter.

VI. In the rejoinder to the statement of grounds of appeal the patent proprietor (respondent) maintained the set of claims as upheld by the opposition division as the main request as well as submitting two sets of claims as auxiliary requests. Experimental data were submitted. In this submission, and in a further one, the arguments of the appellant were disputed.

VII. The Board issued a summons to oral proceedings and a communication.

VIII. Oral proceedings were held before the Board on 6 April 2017.

IX. The arguments of the appellant, insofar relevant for the present decision, can be summarised as follows:

The closest prior art was represented by the process of E2. The distinguishing feature was the nature of the acid used, BF_3 of E2 being replaced by the carboxylic acid of the operative claim. As shown by evidence of the opponent submitted during the opposition procedure, the products obtained by the process of E2 and by that of the patent in suit were essentially the same, meaning there was no technical effect associated with the distinguishing feature. Evidence of the patent proprietor submitted in the rejoinder to the appeal could not establish a technical effect since different conditions to E2 had been employed. The objective problem to be solved with respect to E2 was therefore to provide a further process.

This problem was solved by replacement of the acid. The prior art provided many incentives to do this. E5 related to the preparation of amino-functional siloxanes and presented Lewis acids and Bronsted acids as equivalents. The examples of E5 showed that the use of acetic acid resulted in storage stable products, which was a problem common to E5 and E2. A further similarity between E5 and E2 was provided by the fact that the products could be pumped/filled into containers immediately after mixing. Furthermore, general knowledge of the skilled person, as reflected by E9 and E10, showed that acetic acid promoted not only heterocondensation of polysiloxanes and amino alkoxysilanes, i.e. the reaction in E2 and of the patent in suit but also homocondensation of polysiloxane. E2 presented the BF_3 catalyst employed therein merely as an example of various applicable catalysts. Hence E2 itself provided motivation to replace the exemplified acid catalyst. E2 provided no evidence that BF_3 was any better than other catalysts. On the contrary E2 itself was trying to provide simply a different process to that known in the then existing prior art, which aim was accomplished in E2 by variation of the known catalyst. The step of further varying the catalyst as defined by operative claim 1 merely continued the general approach laid out by E2. Furthermore, as discussed before the opposition division, the process of E2 suffered from the disadvantage of the BF_3 remaining in the polymer which provided a clear reason to seek an alternative catalyst. Further E2 was a patent application so there was inherently an incentive for the skilled person - in this case a competitor - to seek to avoid this subject-matter. A number of decisions of the Boards supported the position that no specific motivation to carry out a particular modification was needed if the objective technical problem was only the trivial one of providing further processes and potential solutions were known from the prior art. In the present case it would furthermore be expected that acetic acid would also work in a similar manner to BF_3 providing a clear route to solve the problem (routine experiments to confirm this expectation or "try and see" approach).

Although E5 did employ different ratios of reactants to those required by operative claim 1, these were presented only as preferable in E5 hence there was no reason not to depart from these and employ different ratios, e.g. such as those within the scope of the operative claim. In any case there was no evidence that by employing the ratios of E5 the product would necessarily be different - this had not been demonstrated by the patentee.

Further motivation to replace the catalyst of E2 by a carboxylic acid for the reaction of interest was provided by the disclosures of E1, E3 and E4.

Alternatively E1 could serve as the closest prior art. The process thereof involved prehydrolysis of the alkoxyaminosilane but there was no evidence that this additional step led to different products to those of the process of the operative claims. The evidence of the examples of E1 was that high viscosity products, i.e. products having high molecular weight, were obtained which was also an indication of a regular structure as required by the patent. Due to the prehydrolysis, the aminoalkoxy silane was not directly reacted. Furthermore the example of E1 used KOH as the catalyst. There was indeed no indication in E1, that, if carboxylic acid were to be used as the catalyst instead, the ratio of reactants as specified in the operative claim should be employed. However, conversely, no technical effect had been shown for any of the distinguishing features of the claimed subject matter over E1. In particular, it had not been shown that omission of the prehydrolysis step of the process of E1 led to any effect in terms of the products. Hence starting from E1, the objective problem was also the provision of a further process. The distinguishing feature of the claimed subject-matter compared to E1 was that in the claimed process there was no complete prehydrolysis. However E1 itself taught that it could be beneficial not to have complete prehydrolysis, and also taught to use acetic acid as the catalyst and to use substoichiometric amounts during the condensation reaction. E1 disclosed a very broad range of ratios of terminal OH groups of the polysiloxane to the amino alkoxysilane and no effect had been shown to arise as a result of the restriction of this ratio as specified in the operative claim. Hence the ratios of reactants as specified in the operative claim were within the general wording of E1 and also could not support an inventive step.

X. The arguments of the respondent, as far as relevant for the present decision, can be summarised as follows:

The introduction of the patent set out the intended end uses. The ratios in claim 1 were designed to ensure that the resulting products were suitable for said uses and in particular were intended to ensure that not all the amino groups reacted.

Closest prior art was E2 which provided similar products. The difference over E2 was not only the catalyst but, as a consequence, also the ratio of carboxylate groups to amine groups, which ratio was inherently not disclosed in E2. Even taking the technical problem as the provision simply of a further process, there was no reason to consult E5, which document in any case, would not reveal the specific ratios required by the operative claim. The references to E9 and E10 were not relevant. E9 referred to a very large number of catalysts and consequently was so general as to be irrelevant. E10 related to a different reaction to that underlying the operative claim. Similarly recourse to E1, E3 and E4 would not provide the necessary teaching, in particular in respect of the ratios of components.

The combination of E2 and E5 was not appropriate. Whilst E2 resulted in a similar product to that obtained by the process of the patent in suit, E5 did not. E5 mentioned a large group of catalysts including BF_3 and carboxylic acids. The teaching of E5 in respect of the amount of acid, i.e. permitting a stoichiometric equivalent or slight excess went against the subject-matter as claimed. Similarly the information relating to the ratios of alkoxysilanes to polysiloxane in E5 was incompatible with that required according to the operative claim. Thus if E2 and E5 were to be combined, a large part of the teaching of E5 would have to be disregarded. Similarly combination of E2 with the teachings of E1, E3 or E4 did not render the claimed subject-matter obvious, either because the ratio of reactants was not revealed, or because the nature of the reaction involved was different to that of E2 and the patent in suit.

Regarding the alternative approach starting from E1, this document was not the closest prior art. The products of E1 were different to those resulting from the process of the patent in suit due to the mandatory prehydrolysis step in D1. The argument relating to the viscosity as an indicator that the same or a similar product was obtained was incorrect, as the viscosity only provided an indication of the chain length but not of the actual constitution of the polymer molecules. Since in the process of E1 the alkoxy groups were removed in a first step by hydrolysis there was no reason, nor was it possible, to define a ratio of these to other reaction components. By the same token, this demonstrated that the product of the process of E1 could not be the same as that of the operative claims. Starting from the two-step process of E1 there was no reason to change it to arrive at a process within the terms of the operative claims, which involved a one-step process. Further in the example of E1 KOH was used as the catalyst and it was not obvious to replace this by a carboxylic acid.

XI. The appellant requested that the decision under appeal be set aside and that the patent in suit be revoked.

XII. The respondent requested that the appeal be dismissed, or alternatively that the patent be maintained on the basis of either of the first or second auxiliary requests, each as filed with the response to the statement of grounds of appeal.

Reasons for the Decision

1. Main Request - inventive step

Both parties provided full reasoning on inventive step starting alternatively from E2 or E1 as the closest prior art. In view of this and of the conclusion reached, the Board finds it appropriate to analyse the two approaches separately.

1.1 Approach based on E2 as the closest prior art.

1.1.1 E2 relates to composition obtainable by reaction of:
(A) one monovalent SiC bound residue with primary, secondary or tertiary amino groups,
(B) an organopolysiloxane and
(C) one or more compounds selected from *inter alia* Lewis acid fluoride compounds (claim 1).

According to claim 2 of E2, the catalyst compound (C) can be BF_3 or an addition compound thereof. In the examples of E2 complexes of BF_3 such as BF_3 methanol or BF_3 dibutyl, diethyl or ethylamine complex are employed.

According to the description (page 1, line 40ff) and claim 3 compound (A) can bear both amino groups and alkoxy groups, corresponding to component (A) of operative claim 1.

1.1.2 Consequently the subject-matter of operative claim 1 is distinguished from the disclosure of E2 by the feature that instead of the Lewis acid catalyst, a carboxylic acid is employed. As a consequence of this it follows that the feature relating to the ratio of carboxylic acid groups to amino groups of the amino silane and the partial conversion of aminosilane (A) into its carboxylate salt are also not satisfied by the process of E2.

1.1.3 The parties disagreed on the formulation of the technical problem which according to the appellant was the provision of a further process to provide the target compounds and according to the respondent should take into account technical effects obtained by means of the distinguishing feature. As even accepting the formulation of the appellant, the Board comes to the conclusion that the objection is not successful, there is no need for the Board to analyse the issue in any further detail and the problem with respect to E2 is formulated as the provision of a further process to provide the target compounds.

1.1.4 According to the operative claims, this problem was solved by the aforementioned replacement of the Lewis acid catalysts of E2 by the carboxylic acid and the further distinctions directly arising as a consequence thereof (see above).

1.1.5 Obviousness

The appellant takes the position that the claimed solution to this technical problem constitutes a "try and see" situation, in which the skilled person would have taken the claimed solution (carboxylic acid as catalyst) into consideration in view of the various teachings of the state of the art, and would, by means of routine experiments, have been able to establish whether the desired result was obtained. In making this argument the appellant took the position that the suitability of carboxylic acids as catalysts for the relevant reaction was known to the skilled person.

(a) It was principally argued that the teaching of E5 would render it obvious that Bronsted acids could be used in place of Lewis acids in the reaction disclosed in E2. E5 relates according to claim 1 to a process for the preparation of organyloxy group terminated polysiloxanes by reacting an OH terminated organopolysiloxane with an alkoxysilane bearing at least two alkoxy groups and which alkoxysilane contains a residue which has at least a secondary or tertiary amino group linked to the Si atom via a bivalent hydrocarbon residue. A Bronsted or Lewis acid is required (feature (C) of claim 1). However as defined in claim 1 of E5 and further explained in the abstract and introduction of E5, in particular page 1, lines 28-37 and page 2, lines 24-29 the aim is to provide organyloxy-terminated polyorganosiloxane. Thus the reaction of interest in E5 is one of end-capping, not chain extension as in the case of the patent in suit and E2. This different aim is reflected by the required proportions of reactants employed in E5. Thus at page 7, line 23ff it is required that the alkoxysilanes are employed in a stoichiometric excess with respect to the terminal silanol groups of the polyorganosiloxanes. Operative claim 1 however requires that this proportion be inversed, i.e. that the terminal silanol groups of the polyorganosiloxane be in excess with respect to the Si-bonded alkoxy groups of the alkoxysilane. Furthermore, E5 is not restricted to the use of carboxylic acids as catalysts but according to claim 1 either Bronsted of Lewis acids can be employed.

Since E5 and E2 pursue different aims i.e. chain endcapping and chain extension respectively, the skilled person seeking to provide a modification of the process of E2 would - unless *ex post facto* considerations were applied - have no reason to consider the teaching of E5.

Even if E5 were to be taken into consideration, the teaching thereof does not reveal the modification of the process of E2 necessary to arrive at the subject-matter claimed. On the contrary, it would be necessary not only to select the nature of the acid from the alternatives disclosed in E5 but also to modify the proportions of reagents employed, thus departing from the teaching of E5.

The appellant argued that there was no evidence that by employing the proportions of reactants taught by E5 in the process of E2 different products would arise, or, put differently, that it had not been demonstrated that the combination of the teachings of E2 and E5 would not provide a solution to the problem of providing a further process with respect to E2. However no proof, e.g. in the form of experimental data, was advanced by the appellant who bore the burden of providing evidence in support of this contention. As a consequence this argument of the appellant has the status merely of an unsupported assertion, which assertion furthermore stands contrary to basic chemical knowledge and experience according to which the relative proportions of reactants employed does exert an influence on the outcome of reactions. Accordingly the combination of E5 and E2 does not render the claimed subject-matter obvious.

(b) E1 was invoked, in a further approach, in combination with E2. However as explained in detail in section 1.2 below, the reaction of E1 differs from that according to E2 and the patent in suit in that a mandatory step is hydrolysis of the alkoxy of the aminoalkoxysilane meaning that, since different reactive species are involved, the process is different to that of E2 and the patent in suit. Furthermore, as a consequence *inter alia* of this difference the ratios of reactants as specified in the operative claim are not taught by E1.
Hence the combination of E2 and E1 does not render the claimed subject-matter obvious.

(c) E3 was also invoked in combination with E2. E3 relates to microemulsions of aminopolysiloxanes which, according to page 3, line 49ff can be prepared by aminoalkylation of polysiloxanes or by copolymerisation of amino group containing silanes with non-ionic mono- or polysiloxanes. The aminosilanes correspond broadly to those as specified in the operative claims. According to the examples of E3, carboxylic acid is employed as the catalyst. However the proportions of reactants does not correspond to that required by the operative claims. Thus in example 2 of E3 a substoichiometric ratio of silanol groups of the polyorganosiloxane to the Si-bonded alkoxy groups of the aminosilane is employed. Analogously for the situation as discussed with respect to E5, reliance on the teaching of E3 requires not only importing the catalyst but also modifying the proportions or reactants away from those as defined in E3 in order to arrive at subject-matter falling within the scope of the operative claim. The consequence is, as discussed above for E5, that the disclosure of E3 does not, in combination with that of E2, render the subject-matter claimed obvious.

(d) A similar conclusion is reached with respect to the disclosure of E4. This document also relates to a process for end-capping polyorganosiloxanes rather than chain extension. Furthermore according to the experimental section of E4 the acetic acid is employed in such amount that it is in slight excess compared to the amino groups of the aminosilane component, meaning that the corresponding feature of operative claim 1 is not satisfied. Thus similarly as for the situation with E5, in order to arrive at the subject matter as now claimed, when starting from E2 and combining this with the teachings of E4, it is necessary to ignore that the respective purposes and aims of E2 and E4 are different and furthermore to selectively adopt only certain parts of the teaching of E4 whilst disregarding others. Thus the combination of the teachings of E2 and E4 likewise does not render the claimed subject-matter obvious.

(e) E9 was also invoked in combination with E2. E2 is restricted to the use of Lewis acid fluoride compounds, in particular BF_3 as the catalyst. E2 contains at page 1, lines 21-22 a reference to E9, which according to E2 gives a comprehensive overview of catalysts for the homocondensation of SiOH-SiOH groups. This is indeed the information which can be derived from E9, passage bridging pages 371 and 372. However, the reaction of interest, i.e. that which occurs in the process of the operative claims and in E2 is a heterocondensation of polysiloxane und alkoxyaminosilane and not the homocondensation of organylsilanols as discussed in the cited passage of E9. Thus neither E9 directly nor the discussion of this document in E2 provides any hint that carboxylic acids would be effective catalysts in the heterocondensation reaction of interest.

As a result of the incompatibilities or "mismatch" between the the reaction of E2 and the patent in suit and teachings of E9 regarding the utility or activity of the catalysts in terms of the reactions promoted, i.e. the absence of any connection to the reaction of interest in E9, the "try and see" situation, put forward by the appellant in particular with respect to E9 and E10 (discussed in the following section), i.e. involving routine experiments in a situation where a particular course of action was indicated by the prior art, does not apply. Consequently the corresponding arguments and case law invoked by the appellant in this connection are also not of relevance.

(f) A similar conclusion is reached with respect to E10. This document lists in column 5, starting at line 15, silanol condensation catalysts and gives a large number of possible classes of compounds, including organic acids, acetic acid being one of those explicitly mentioned. The reaction which is being catalysed in E10 is however that between ethylene and an unsaturated organosilane compound, not the reaction of chain extension which is the subject-matter of E2 and the patent in suit. Furthermore there is no indication in E10 - either explicit or by implication - which would suggest any equivalence between the Lewis acids as employed in E2 and organic acids as specified according to the operative claims in any context, let alone specifically in the context of the chain-extension reaction of E2 and the patent in suit. Accordingly, E10 can also provide no basis for the position that it would be obvious to replace the catalysts of E2 by those defined in the operative claim in order to provide a further process to that of E2. As a consequence of the incompatibilities and differences between the teachings of E2 and E10, the "try and see" or routine experimentation argument which was also invoked by the appellant with respect to E10 does not succeed, analogously for the situation with respect to E9.

1.1.6 The conclusion is therefore that starting from E2 none of the combinations with other documents E1, E3, E4, E5, E9 or E10 proposed by the appellant would lead in an obvious manner to the subject-matter as now claimed, even in the context of solving the problem of providing only an alternative process to that of E2.

1.2 Approach based on E1 as the closest prior art

1.2.1 E1 is directed, according to the introduction in column 1, to the synthesis of zwitterionic siloxane polymers. The starting point of the invention of E1 is represented by products obtained by reaction of aminoalkyldimethoxysilanes with low molecular weight polydimethylsiloxanes. The known products have insufficient molecular weight and the aim of E1 is to address this aspect. E1 reports in column 2, starting at line 4 that high molecular weight aminoalkyl-siloxane polymers were difficult to obtain from polymerisation of aminoalkyl-dimethoxysilanes and hydroxy-terminated polydimethylsiloxane oligomers by the then known methods. In particular it is stated (column 2, lines 9-12):

"It is difficult to obtain high molecular weight aminoalkyl siloxane polymers where the difunctional aminoalkyl-silane has alkoxy or aryloxy functional groups".

This problem is addressed, as set out in the subsequent passage of E1, by subjecting the functional groups of the aminoalkyl-silane to prehydrolysis, providing a route to the desired high molecular weight products. According to column 3, line 42ff hydrolysis can be accomplished by exposing the aminoalkyl silanes to water, base or acid, including certain named carboxylic acids.

Due to the prehydrolysis of the aminoalkyl silane, with the express purpose of removing the alkoxy groups, the reactive species in the process of E1 is not, as required by the operative claims, "an amino silane... which contains...at least one alkoxy group bonded to Si".

1.2.2 From the foregoing it emerges that the process of E1 is fundamentally different to that of the patent in suit. Indeed, according to the teaching of E1 the process of the patent in suit is not possible or not efficient for providing high molecular weight products due to the presence of the silicon bonded alkoxy groups which are a mandatory feature of operative claim 1.

It follows that other features of the operative claim, in particular the ratio of silanol groups to Si-bonded alkoxy groups of the amino silane are not satisfied by the process of E1 and even would be contrary to the teaching thereof.

1.2.3 Regarding the argument of the appellant that there was, in the case of E1, no evidence that the prehydrolysis resulted in different products, it was for the appellant to provide evidence to support this contention. The reference to the high viscosity of the resulting products reported in E1 is not sufficient to discharge this burden, since the viscosity gives only very general information about the product, i.e. an indication of the total chain lengths/molecular weight, but does not given any insight - in this case - to the actual structure of said polymer chains. As in the situation considered with respect to E2 and E5 above, the normal expectation in chemistry would be that employing modified reactants would lead to different products, meaning that the contention is in any case inconsistent with what the skilled person would expect. By the same token, the contention that E1 requires only partial hydrolysis, which contention appears to be based on an interpretation of the passage at column 3, lines 44-46 of E1 reading "Where hydrolysis of all the alkoxy/aryloxy functional groups is desired...." does not change this analysis, since even partial hydrolysis would result in different reactive species being - at least in part - involved in the reaction meaning that the general expectation would be that different products would result. The only way in which this passage of E1 would lead to a process - potentially - corresponding to that claimed would be to interpret it as meaning that hydrolysis could be omitted entirely, which interpretation would be inconsistent with the explicit teaching of E1 as discussed above, and hence "obvious" only on the basis of knowledge of the process of the patent in suit, i.e. *ex post facto.*

1.2.4 It is therefore concluded that in order to arrive at the subject-matter claimed from the teaching of E1, it would be necessary to discard the central teaching of E1, namely the need for prehydrolysis of the alkoxysilane.

1.2.5 For this reason the disclosure of E1 cannot provide a route to the claimed process and does not render the subject-matter claimed obvious.

1.3 In conclusion neither of the approaches proposed by the appellant lead to the conclusion that the subject-matter claimed is obvious.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

Beschwerdekammern des Europäischen Patentamts
Boards of Appeal of the European Patent Office
Chambres de recours de l'Office européen des brevets

B. ter Heijden D. Semino

Decision electronically authenticated