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D E C I S I O N
of 20 February 2003

Case Number: T 0299/01 - 3.3.5

Application Number: 91913688.7

Publication Number: 0495039

IPC: C01B 33/193

Language of the proceedings: EN

Title of invention:
Silicas

Patentee:
INEOS Silicas Limited

Opponent:
DEGUSSA AG

Headword:
Silicas/INEOS

Relevant legal provisions:
EPC Art. 123(2), 54, 56

Keyword:
"Novelty (yes)"
"Inventive step (yes)"

Decisions cited:
G 0010/91

Catchword:
-

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Case Number: T 0299/01 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 20 February 2003

Appellant: (Opponent)
DEGUSSA AG
Benningsplatz 1
D-40474 Düsseldorf (DE)

Representative:
Weber, Wolfgang, Dr
Degussa AG
Intellectual Property Management
Patente und Marken
Standort Hanau
Postfach 1345
D-63403 Hanau (DE)

Respondent: (Proprietor of the patent)
INEOS Silicas Limited
Bank Quay
Warrington WA5 1AB (GB)

Representative:
Jackson, John Derek
Crosfield Limited
Warrington
Cheshire WA5 1AB (GB)

Decision under appeal: **Interlocutory decision of the Opposition Division of the European Patent Office posted 9 January 2001 concerning maintenance of European patent No. 0 495 039 in amended form.**

Composition of the Board:

Chairman: R. K. Spangenberg
Members: M. M. Eberhard
H. Preglau

Summary of Facts and Submissions

I. The appeal is from the decision of the opposition division according to which European patent No. 0 495 039 in amended form meets the requirements of the Convention. The patent was granted in response to European patent application No. 91 913 688.7. The decision under appeal was based on the granted claims, as the main request, and on the amended claims filed on 6 October 2000 as the auxiliary request. Claim 1 of the auxiliary request reads as follows:

"1. An amorphous precipitated silica having:
i) a surface area in the range from 250 to 600 m^2/g,
ii) an oil absorption (using linseed oil) from 90, preferably from 110 to 160 $cm^3/100g$,
iii) a weight mean particle size in the range from 5 to 20 microns,
iv) a plastics abrasion value in the range from 8 to 14,
v) a transmission of at least 80% within the refractive index range of 1.435 to 1.444, and
vi) a mean pore diameter in the range from 1.5 to 21 nm."

II. During the opposition proceedings the appellant (opponent) relied *inter alia* on EP-A-0 227 334 (D1) and EP-A-0 308 165 (D2). The opposition division took the view that the subject-matter of claim 1 as granted lacked novelty over the disclosure of D1. The silicas of claim 1 according to the auxiliary request were new with respect to D1. The cited prior art did not disclose silicas having a transmission of 80% in combination with the pore structure, abrasive properties or specific surface area as defined in claim 1. It was plausible that such silicas left

considerably more room for variations of transparent toothpaste formulations. Furthermore, the prior art contained no enabling teaching as to how to prepare such silicas.

III. Oral proceedings took place on 20 February 2003. The appellant requested that the decision under appeal be set aside and that the patent be revoked. The respondent (proprietor of the patent) requested that the appeal be dismissed.

IV. The appellant's arguments can be summarised as follows:

Claim 1 did not meet the requirements of Article 123(2) EPC since the upper abrasion value of 14 was not disclosed in the PCT application in combination with a refractive index of 1.444. It was only disclosed in Example 3 of the PCT application but with a refractive index of 1.452 which fell outside the claimed range.

The claimed subject-matter was not novel with respect to D1 which disclosed precipitated silicas having values of surface area, oil absorption, mean particle size and abrasion all falling within the claimed ranges (see Examples 3 and 4). These silicas had a transmission of at least 70% in the refractive index range of 1.440 to 1.445. The expression "at least 70%" encompassed values of at least 80%. Furthermore the terms "translucent" and "transparent" were defined in D1 and in the patent in suit in the same way. In addition the production method given in D1 was identical to that disclosed in the patent in suit and thus the resulting products were also identical. The difference in the operating conditions, in particular the temperature, was not so substantial as to result in a different product. The examples of the patent in suit showed that very different temperatures led to silicas with similar transmission values. The claimed silicas

also lacked novelty over the silicas of D2 which exhibited abrasion values of about 15. This wording was vague and encompassed values of 13 or 14. The abrasion value of 14 was disclosed in D1 taking account of the margins of error of the measurement method.

The technical problem of the patent in suit was to provide silicas suitable for use in transparent toothpastes and having a medium degree of abrasivity. D1 represented the closest prior art. The silicas of D1 were suitable for use in toothpastes and had high values of oil absorption which could be expected to give more structure to the toothpaste formulations. As the values of oil absorption in D1 and in the patent in suit lay within the same range, the said problem was already solved by D1. The abrasion value of the silicas in Examples 3 and 4 of D1 were also identical to the claimed ones and the transmission values were obvious in view of D1 disclosing values of at least 70%. The claimed silicas also did not involve an inventive step in view of the teaching of D2. A transmission of at least 70% at a refractive index of 1.444 was disclosed in D2 in combination with surface areas, particle sizes, and pore diameters falling within the claimed ranges. The technical problem was already solved by the disclosure of an abrasion value of about 15 in D2, which encompassed the value 14 taking account of the measurement accuracy. The wording "about 15 to about 28" suggested that there was no clear boundary for the abrasion. Therefore, it was obvious to choose a range below 15.

V. The respondent presented *inter alia* the following arguments:

The objection under Article 123(2) had not been raised in the opposition proceedings and was not allowable. The respondent did not agree for this new ground of

opposition to be introduced into the proceedings. The claimed silicas were novel over those of D1 since there was no disclosure of a transmission of at least 80% in D1 and the silicas of D1 were suitable for opaque or translucent toothpastes, not for transparent formulations. The production method of the patent in suit differed from that given in D1 at least by the thorough mixing described in the patent in suit. A comparison of the reaction conditions in Examples 3 and 4 of D1 with those of Examples 1 and 3 of the patent in suit demonstrated that the electrolyte/silica ratio in the patent was much higher than in D1 and the reaction temperature was lower. Both the said ratio and the reaction temperature were important parameters in determining silica properties. The products resulting from the process of the patent in suit thus differed from those of D1. D2 did not take away the novelty of the claimed silicas. The results of the abrasion test were expressed as the nearest integer and the wording "about 15" did not mean more than that. The skilled person would have interpreted the expression "about 15" as a value between 14.5 and 15.5. The accuracy of the measurement was expected to be of ± 0.5 when using standardised samples and repeating the test five times. Furthermore, there was no evidence that the silicas of D2 could have a transmission above 80%, except at refractive index values well removed from 1.444. The silicas of Examples 3 and 4 of D1 represented the closest prior art. The technical problem was to provide a silica having a medium degree of abrasivity and being suitable for use in transparent toothpastes. The silicas of D1 were not suitable for use in transparent toothpastes and D1 accordingly did not teach which reaction conditions to use in order to solve the said problem. The claimed silicas had a high transmission at lower refractive indices than those of D2, thus resulting in cheaper toothpaste formulations. D2 contained no information suggesting that an abrasion

value below 15 was possible, let alone in combination with a high transmission at lower refractive indices and did not disclose how this could be achieved.

Reasons for the Decision

1. The appeal is admissible.

2. Amended claim 1 differs from claim 1 as granted only by the transmission value of at least 80% instead of at least 70%. The PCT application discloses a transmission of at least 70%, preferably at least 80%, within the refractive index of 1.435 to 1.456 in combination with a surface area in the range from 250 to 600 m^2/g, an oil absorption of 90 to 160 $cm^3/100g$, a weight mean particle size in the range from 5 to 20 microns, a plastic abrasion value in the range from 8 to 15 and a mean pore diameter in the range from 1.5 to 21 nm: see page 3, first paragraph. The range of 1.435 to 1.444 is disclosed on page 4, second paragraph. Examples 2, 7 and 8 on page 17 of the PCT application show that the transmission may be of at least 80% at a refractive index within the said range of refractive index. Therefore, the amendment introduced after grant meets the requirement of Article 123(2) EPC. This was not disputed by the appellant. The protection conferred by the amended claims 1 is clearly restricted with respect to that of the granted claims.

For the first time at the oral proceedings the appellant argued that the upper abrasion value of 14 was not disclosed in the PCT application in combination with a refractive index of 1.444, and thus that claim 1 did not meet the provisions of Article 123(2) EPC. The board observes in this respect that an abrasion value of above 14, and thus the value 14 itself, is indicated

on page 4, last paragraph, of the PCT application and a refractive index range of 1.435 to 1.444 is stated on page 4, 2nd paragraph, thereof. However, the question whether or not the combination is disclosed in the PCT application has to remain open for the following reasons. Although both ranges for the abrasion and the refractive index values (from 8 to 14 and from 1.435 to 1.444 respectively) were already indicated in granted claim 1, no objection under Article 100(c) EPC had been raised by the appellant in his notice of opposition or during the opposition proceedings. Furthermore, the appellant's objection does not arise out of the amendment introduced in claim 1 during the opposition proceedings. Therefore, the appellant's objection amounts to raising a new ground of opposition. According to opinion G 10/91 (OJ EPO, 1993, 420), fresh grounds of opposition may be considered in the appeal proceedings only with the approval of the patentee. The respondent's representative having refused to give his agreement, the matter is not further taken into consideration by the board.

3. Concerning the issue of novelty, the appellant's arguments that the claimed silicas lack novelty over the disclosure of D1 cannot be followed for the following reasons. As pointed out by the appellant, D1 discloses in Examples 3 and 4 two amorphous precipitated silicas having a surface area, an oil absorption, a weight mean particle size and an abrasion value falling within the ranges defined in claim 1 on file. At the oral proceedings before the board, the respondent confirmed that he accepted the opposition division's assumption that the pore diameter lies within the range stated in claim 1. Therefore, novelty of the claimed subject-matter depends on whether or not the silicas of Examples 3 and 4 of D1 have a transmission of at least 80% within the range of refractive index of 1.435 to 1.444. According to D1,

the transmission (589 nm) of the silicas according to the invention of D1 is at least 60% in the range of refractive index 1.438 to 1.448 and at least 70% in the range 1.440 to 1.445. These transmissions show the silicas are of value for opaque and translucent dentifrices but not the commercially used transparent formulations (see page 2, lines 49 to 52). Typical formulations using the silicas of the invention in D1 are listed on page 6 (see page 5, lines 51 to 52, whole page 6). These formulations are those of an "opaque, white toothpaste" and a "translucent gel toothpaste". Therefore, the statement on page 2 that the silicas are suitable for use in opaque and translucent dentifrices is confirmed on pages 5 to 6 of D1. The respondent argued that there was a mistake in the transmission values given on page 2 of D1 since a silica with a transmission of at least 70% would be suitable for a transparent toothpaste whereas the silicas of D1 were stated to be suitable for opaque and translucent toothpastes. According to the respondent a transmission below 70% would be consistent with the use in opaque or translucent toothpastes indicated in D1. In the board's view the value of at least 70% stated on page 2, line 50, is not consistent with the fact that the silicas are suitable for use in opaque toothpastes since the transmission value is well-known to be below 70% in this case. Even if it were assumed in favour of the appellant that the transmission value at the borderline between translucent and transparent toothpastes is not exactly 70% but might be set up slightly higher by other manufacturers, the situation is, however, quite different in the case of a transmission value of at least 80% as now stated in claim 1. It was not disputed by the appellant that silicas having transmission values of at least 80% within a particular refractive index range are usually considered to be suitable for use in transparent toothpaste formulations. Taking into account that the

silicas of D1 are said to be suitable for opaque and translucent dentifrices, thus implying that their transmission within a particular range of refractive index is lower than 80%, the board is not convinced that it can be directly and unambiguously derived from the disclosure on page 2, line 5, of D1 that the silicas of Examples 3 and 4 have a transmission of at least 80% in the range of refractive index 1.435 to 1.444.

3.1 The appellant's arguments that the silicas of D1 were prepared by the same process as those of the patent in suit, thus leading to the same products, cannot be followed by the board. Firstly, it should be noted that according to the patent in suit mixing is an important feature in the reaction of silicate and sulphuric acid. Accordingly specific mixing conditions are described on page 5, lines 48 to 55, of the patent in suit. On the contrary, D1 is fully silent in this respect and does not contain any information concerning the mixing step. Furthermore, it is correct that the general process disclosed on page 5, lines 27 to 41, of D1 is similar to that defined at page 6, lines 5 to 16, of the patent in suit. However, the two processes are incompletely defined in these passages since the reactant concentrations, the reactant volumes and the reaction temperatures are not indicated and both D1 and the patent in suit make reference to the values given in Table 1 for these parameters. A comparison of Table 1 of D1 with Table 1 of the patent in suit shows that the silicas were prepared differently. More precisely a comparison of Examples 3 and 4 of D1 with Examples 1 and 3 of the patent in suit respectively, which were prepared on the same scale (same vessel capacity) using the same electrolyte, demonstrates that the electrolyte/silica ratio used in the examples of the patent in suit is much higher than that used in Examples 3 and 4 of D1. It is credible that this ratio

is an important parameter in determining the silica properties as pointed out by the respondent and not contested by the appellant. Furthermore the temperature was maintained at 98°C during the addition of the reactants in D1 whereas it was maintained at 80 or 85°C in the examples of the patent in suit. Therefore the temperature difference was of 13 or 15°C. As pointed out by the appellant, a temperature of 98°C was indeed used in Example 2 of the patent in suit, however this example cannot be compared with Example 3 of D1 because of the different electrolyte used in these examples (sodium sulphate instead of sodium chloride). The appellant's assertion that the temperature difference is not so substantial as to lead to different products is not convincing without evidence supporting this assertion for the following reasons. First, it is well-known in this technical field that small differences in the method of preparation can lead to products with different properties. Furthermore it is disclosed in D2, which also discloses the preparation of amorphous precipitated silicas by a comparable method, that the reaction temperature is critical on the transmission and pore diameter of the resulting silica as shown by comparative Example 7. This example shows that by using a temperature of 60°C instead of a temperature within the range 45 to 55°C the maximum transmission is considerably reduced and lies well outside the desired range of at least 70% in the refractive index range of 1.444 to 1.460 (see D2, page 3, lines 22 to 35; page 6, lines 52 to 53; page 7, Table I; page 8, Table II). Under these circumstances the respondent's mere assertion that the temperature difference would not lead to a different product cannot be accepted, all the more so as the burden of proof rests on him in this respect.

The appellant's further argument that Examples 1 and 3 of the patent in suit showed that a temperature difference of 5°C at 80°C (ie 80°C and 85°C respectively) had no influence on the transmission value, is also not convincing since these two examples were carried out at a very different scale so that no conclusion can be drawn as to the influence of the temperature. Furthermore, the temperature difference between the compared examples of D1 and those of the patent in suit is not 5°C, but 13 or 15°C. For the preceding reasons, the board considers that the claimed silicas are novel with respect to those of D1.

3.2 D2 discloses amorphous precipitated silicas having a BET surface area, a weight mean particle size and a mean pore diameter which all fall within the claimed ranges. They exhibit perspex abrasion values from about 15 to about 28, preferably to about 25, and a transmission of at least about 70% in the refractive index range of 1.444 to 1.460 (see claim 1). Table II of D2 (see pages 9 and 10) shows that the oil absorption of these silicas, except the silica of Example 4, also falls within the claimed range. The appellant alleged that the vague wording "about 15" used to define the lower limit of the abrasion encompassed the value 14 and that the value of 14 would be disclosed in D1 in view of the margin of error of the measurement method. This was contested by the respondent who, on his side, argued that this expression would be understood as meaning a value between 14.5 and 15.5. According to the respondent the test methods used in D2 and in the patent in suit for determining the abrasion were exactly the same and the abrasion was in both cases expressed as the nearest integer; the wording "about 15" would reflect this fact and nothing more. Concerning the accuracy of the test method, the inventor of the patent in suit, who is also one of the inventors in D1, indicated at the oral

proceedings that the accuracy would be expected to be of ± 0.5 when effecting a standardisation with known samples and repeating the test five times. The board observes that the expression "about 15" is not further defined in D2. The abrasion values of all the exemplified silicas (examples and comparative examples) are from 17 to 25, ie well above the value 15. The board cannot find any information in D2 from which it would be directly and unambiguously derivable that the expression "about 15" might mean a value as low as 14, all the more so as the purpose of D2 is to provide silicas having high levels of abrasivity with good transparency when incorporated into a dentifrice formulation (see page 2, lines 49 to 54). The respondent's arguments concerning the accuracy of the measurement method were not contested by the appellant so that the wording "about 15" cannot, on the basis of measurement accuracy, be considered as taking away the novelty of the claimed range. Furthermore, the fact that expression "about 15" might encompass values lower than 15 does not mean that the value 14 is disclosed. Under these circumstances, the board has doubts that D2 discloses silicas having an abrasion value of 14 in combination with the BET surface, particle size and pore diameter stated in claim 1 thereof. The board further notes that D2 would destroy the novelty of the claimed silicas only if a silica having both an abrasion value of 14 and a transmission of at **least 80%** at the refractive index of **1.444** were disclosed in D2. However, according to D2 the silicas exhibit a transmission of at **least 70%** in the refractive index range of **1.444 to 1.460**. Table II on pages 8 and 9 shows that when the transmission is 79.5% or greater the refractive index varies from 1.45 to 1.4581 (see Examples 1, 4, 5, 8, 10 and 11). The two comparative Examples 7 and 9 in which the refractive index is closer to the limit of 1.444 (1.446 for Example 7

and 1.4426 for Example 9) the transmission is very low, namely 53.5% and 55% respectively. It is not directly and unambiguously derivable from this teaching that the silicas of D2 can have a transmission of at least 80% at a refractive index of 1.444. Under these circumstances the appellant's allegation that D2 discloses silicas having a transmission of at least 80% at a refractive index of 1.444 cannot be accepted in the absence of evidence to support this allegation. For the preceding reasons, the board comes to the conclusion that the claimed subject-matter is new over the disclosure of D2. It is also novel with the respect to the third document cited during the opposition proceedings. This was not in dispute.

4. D1 represents the closest prior art, in particular Examples 3 and 4 thereof. As already indicated above, the silicas of D1 are suitable for use in opaque or translucent toothpastes. Starting from D1, the technical problem underlying the patent in suit can be seen in the provision of silicas which are suitable for use in transparent toothpaste formulations. It is proposed to solve this problem by silicas having the combination of properties as stated in claim 1. They differ from the silicas of D1 in particular by having a transmission of at least 80% within the refractive index range of 1.435 to 1.444 in combination with the other parameters defined in claim 1. In view of the examples in the patent in suit, it is credible that this problem has actually been solved by the claimed silicas.

4.1 Although it would appear to be obvious that the silicas of D1 have to be modified such that their transmission is high (ie ≥80%) within a certain range of refractive index in order for them to be suitable for use in transparent toothpaste formulations, D1 does not contain any information suggesting that it would be

possible to provide silicas having a transmission of at least 80% within the refractive index range of 1.435 to 1.444 in combination with the additional properties defined in claim 1. D1 discloses a process for manufacturing silicas having the characteristics given in D1, however it could not be inferred therefrom how the process conditions had to be changed to arrive at silicas having the claimed combination of characteristics. The appellant made reference to the teaching on page 2, lines 37 to 38, of D1 in support of his arguments concerning lack of inventive step. It is disclosed in this passage that "the high values of oil absorption can be expected to give more structure (liquid thickening effect) to the formulations containing the precipitated silica abrasives of the invention". It is not clear to the board how this teaching about the high oil absorption and the resulting liquid thickening effect would have been of assistance to the skilled person confronted with the problem of transparency stated above. No further explanation was given by the respondent in this respect. In any case this passage would not have given any indication towards the solution proposed by the patent in suit.

4.2 The appellant's second line of arguments concerning the lack of inventive step with respect to the teaching of D2 is not convincing for the following reasons. Taking into account that D2 discloses amorphous precipitated silicas suitable for use in transparent toothpaste compositions, the board can accept that D2 is also an appropriate starting point for the assessment of inventive step although it concerns silicas having a high instead of a medium degree of abrasivity. Starting from D2, the technical problem to be solved can be seen in the provision of other silicas suitable for use in transparent toothpaste formulations. It is credible in view of the examples of

the patent in suit that this problem has actually been solved by the silicas having the combination of characteristics defined in claim 1. This was not disputed.

The silicas of D2 have inter alia a high degree of abrasivity, ie about 15 to about 28, and a transmission of at least about 70% in the refractive index range of 1.444 to 1.460, which makes them suitable for use in transparent toothpaste compositions. They are produced by a process comprising a precipitation in the temperature range of about 45°C to about 55°C, and wherein the electrolyte/silica weight ratio is from about 0.4:1 to about 1.2:1. This temperature range is critical on the transmission and pore diameter of the resulting silica (see page 2, lines 4 to 5 and 49 to 54; page 6, lines 51 to 53; claims 1 and 10). It cannot be inferred from D2 that it would be possible to obtain silicas still exhibiting the pore diameter, the BET surface area and the particle size stated therein while simultaneously having a lower abrasion value (ie within the range 8 to 14) and a transmission of at least 80% in the refractive index range of 1.435 to 1.444. This document is accordingly silent as to how the process disclosed in D2 should be modified in order to arrive at the claimed silicas. The lower value of about 15 for the abrasion in D2 would not have suggested how silicas having lower abrasion values of from 8 to 14 and a transmission value of at least 80% in the refractive range of 1.444 to 1.460 might be prepared, the other properties remaining as stated in D2. In this context the board refers in particular to the comparatives Examples 6, 7 and 9 of D2 which show that the maximum transmission at refractive index values of 1.432, 1.446 or 1.4426, ie values within the

claimed range or very close thereto, is relatively low (ie 38.3%, 53.5% or 55%) when increasing the ageing time or performing the precipitation at a temperature of 60°C instead of 45 to 55°C.

4.3 The third document cited by the appellant during the opposition proceedings was not relied upon at the appeal stage. Its disclosure likewise cannot point towards the claimed silicas, even when considered in combination with the preceding documents. It follows from the above that the silicas as defined in claim 1 meet the requirement of inventive step set out in Articles 52(1) and 56 EPC.

5. Claim 1 being allowable, the same applies to dependent claims 2 to 11, whose patentability is supported by that of claim 1. The toothpaste composition according to claim 12 contains 5 to 50% by weight of the precipitated silica defined in any of the preceding claims. Therefore its patentability also derives from that of claim 1.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

U. Bultmann R. Spangenberg

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