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**Datasheet for the decision
of 27 November 2009**

Case Number: T 1743/06 - 3.3.05

Application Number: 96918686.5

Publication Number: 0835223

IPC: C01B 33/00

Language of the proceedings: EN

Title of invention:

Amorphous silicas and oral compositions

Patentee:

INEOS Silicas Limited

Opponent:

RHODIA CHIMIE

Headword:

Amorphous silica/INEOS

Relevant legal provisions:

EPC Art. 83, 100(b)

EPC R. 42(1)(e)

Relevant legal provisions (EPC 1973):

-

Keyword:

"Sufficiency of disclosure (no): crucial process feature missing and insufficient guidance for performing the invention without undue burden over the whole range claimed"

Decisions cited:

T 0014/83, T 0409/91, T 0435/91

Catchword:

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Case Number: T 1743/06 - 3.3.05

D E C I S I O N
of the Technical Board of Appeal 3.3.05
of 27 November 2009

Appellant: INEOS Silicas Limited
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 27 October 2006
revoking European patent No. 0835223 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: G. Rath
Members: J.-M. Schwaller
S. Hoffmann

Summary of Facts and Submissions

- I. This appeal was lodged by the patentee (hereinafter "the appellant") against the decision of the opposition division revoking European patent 0 835 233.

In the contested decision, the opposition division concluded that claims 1 and 18 of the fifth auxiliary request, which read as follows:

"1. Amorphous silica characterized by:

- an RDA value between 40 and 70,*
- an oil absorption between 100 and 145 cm³/100g,*
- a BET surface area of 50 to 200 m²/g*
- and a peak of light transmission and a light transmission of more than 70%, measured in a range of sorbitol/syrup mixtures, in the refractive index range of 1.445 to 1.456."*

18. Process for the production of amorphous silicas according to claims 1 to 17 comprising:

- adding a 17.0 to 21.5% solution of 2.1 to 2.5 Molar Ratio silicate solution to water,*
- then further adding a 17.0 to 21.5% solution of 2.1 to 2.5 Molar Ratio silicate solution together with a 15 to 20% sulfuric acid solution, over a period of over 40 minutes at such flow rates that the pH is maintained in the range from 8.0 to 9.0,*
- then aging the resultant slurry for a period of 0 to 30 minutes at a temperature of 90 to 100°C,*
- doing a second addition of a 15 to 20% sulfuric acid solution to bring the pH down to pH 3 to 5,*

- *aging the resulting slurry for a period of 0 to 20 minutes at pH 5 at a temperature of between 90 and 100°C,*
- *adjusting the pH to pH 3.5 to 5, and*
- *eventually filtering, washing and drying the final slurry."*

infringed the requirements of Article 83 EPC, because in the patent in suit any data was missing concerning:

- (a) the amount of silica used in the measurement of the light transmission of silica in sorbitol/water mixtures

and

- (b) the stirring speed during the preparation of the silica claimed,

so that the invention could not be carried out in a manner sufficiently clear and complete by a person skilled in the art.

II. With its statement setting out the grounds of appeal, the appellant submitted four amended sets of claims as main and first to third auxiliary requests, respectively. It also submitted several documents, among which:

D10: Chemineer, Inc., Reprint from Chem. Eng.,
26 April 1976, pages 102 to 110, and subsequent excerpts referred to by this excerpt.

- III. In a communication accompanying the summons to oral proceedings, the board expressed its provisional opinion that in the absence of strong evidence that the features (a) and (b) indicated above could be arrived at without undue burden by a person skilled in the art, there were strong doubts that the requirements of Article 83 EPC would be fulfilled.
- IV. Under cover of a letter dated 1 October 2009, the appellant submitted four new requests in replacement of those then on file.
- V. At the oral proceedings, which took place on 27 November 2009, after an initial discussion which concerned essentially the disclosure of the invention, the appellant dropped the four requests on file and submitted two new sets of claims as main and auxiliary requests, respectively, with independent claims 1 and 12 of said requests reading as follows:

Main request:

*"1. Amorphous silica characterized by:
an RDA value between 40 and 70,
an oil absorption between 100 and 145 cm³/100g,
a BET surface area of 50 to 200 m²/g,
a structural water content of between 3.5% and 5.0%,
a pH in 5% solution of between 6 and 7.5,
a loose bulk density of between 200 and 300 g/l,
a skeletal density of above 2.1 g/cm³, and
a moisture loss of less than 7% w/w at 105°C,
a mercury pore volume of above 1 cm³/g,
a mean pore diameter between 25 and 100 nm,*

wherein the silica has a peak in light transmission, measured in a range of sorbitol/syrup mixtures, in the RI range of 1.445 to 1.456."

"12. Visually clear toothpaste composition having a refractive index of above 1.445 and comprising 5 to 25% by weight of an amorphous silica as claimed in any one of claims 1 to 11, said toothpaste composition having an RDA of 30 to 60."

Auxiliary request:

*"1. Amorphous silica characterized by:
an RDA value between 40 and 70,
an oil absorption between 100 and 145 cm³/100g,
a BET surface area of 50 to 200 m²/g,
a structural water content of between 3.5% and 5.0%,
a pH in 5% solution of between 6 and 7.5,
a loose bulk density of between 200 and 300 g/l,
a skeletal density of above 2.1 g/cm³, and
a moisture loss of less than 7% w/w at 105°C,
a mercury pore volume of above 1 cm³/g,
a mean pore diameter between 25 and 100 nm."*

"12. Visually clear toothpaste composition having a refractive index of above 1.445 and comprising 5 to 25% by weight of an amorphous silica as claimed in any one of claims 1 to 11, said toothpaste composition having an RDA of 30 to 60."

VI. The appellant requested that the decision under appeal be set aside and that the patent be maintained on the basis of the claims according to the main request filed at the oral proceedings on 27 November 2009, or in the

alternative, according to the auxiliary request, also filed on 27 November 2009.

The respondent requested that the appeal be dismissed.

Reasons for the Decision

1. Sufficiency of disclosure

1.1 According to Article 83 EPC and its counterpart in Article 100 b) EPC, the requirement of sufficient disclosure means that an invention shall be disclosed in a manner sufficiently clear and complete for it to be carried out by a person skilled in the art.

The specific requirement of Rule 42(1)(e) EPC that the description shall describe in detail at least one way of carrying out the invention claimed is, according to established jurisprudence, sufficiently fulfilled only if:

- (i) the disclosure allows the invention to be performed in the whole range claimed (T 409/91, OJ 1994, 653, point 3.5 of the reasons; T 435/91, OJ EPO 1995, 188, point 2.2.1 of the reasons),
- (ii) the whole subject-matter as defined in the claims can be carried out by the person skilled in the art, at the filing date of the application, without undue burden (see e.g. decision T 14/83, OJ EPO 1984, 105, point 6 of the reasons).

1.2 In the case at issue, claims 1 of both requests are directed to an amorphous silica characterised by 10 and 11 different parameters, respectively, and the contested patent describes in detail the preparation of two specific amorphous silicas having parameter values falling within the claimed ranges (see examples 1 and 2).

The board however observes that the definition "amorphous silica" comprises a host of possible chemical compounds which may or may not satisfy the multiplicity of parameters defined in the claims of the requests at issue and in this context, the question arises whether the patent contains sufficient information about how these parameters are to be reliably achieved so that the person skilled in the art has at his disposal a process which leads him in a direct way to the amorphous silicas claimed.

1.3 Concerning the preparation of the amorphous silicas disclosed in the patent in suit, there is the information at paragraphs [0022] and [0021] that amorphous silicas presenting good cleaning characteristics without damaging teeth and which are particularly good at preventing stain formation can be obtained through a process *"comprising:*

- *adding a 17.0 to 21.5% solution of 2.1 to 2.5 Molar Ratio silicate solution to water,*
- *then further adding a 17.0 to 21.5% solution of 2.1 to 2.5 Molar Ratio silicate solution together with a 15 to 20% sulfuric acid solution, over a period of over 40 minutes at such flow rates that the pH is maintained in the range from 8.0 to 9.0,*

- *then aging the resultant slurry for a period of 0 to 30 minutes at a temperature of 90 to 100°C,*
- *doing a second addition of a 15 to 20% sulfuric acid solution to bring the pH down to pH 3 to 5,*
- *aging the resulting slurry for a period of 0 to 20 minutes at pH 5 at a temperature of between 90 and 100°C,*
- *adjusting the pH to pH 3.5 to 5, and*
- *eventually filtering, washing and drying the final slurry".*

The board however notes that the description of the contested patent does not give any details as to how the above process conditions "for preparing amorphous silicas presenting good cleaning characteristics without damaging teeth and which are particularly good at preventing stain formation" might be modified in order to achieve reliably the parameters of the specific amorphous silicas defined in the claims 1 at issue.

- 1.4 Having been questioned on that point at the oral proceedings, the appellant admitted that by carrying out the process according to paragraphs [0022] and [0021] of the contested patent, the skilled practitioner would not necessarily arrive at an amorphous silica falling within the wording of claims 1 of both requests at issue. It however contended that by varying the process conditions described in detail with respect to the two amorphous silicas specifically exemplified in the contested patent, one would arrive at the preparation of amorphous silicas falling within the ambit of the claims 1 of both requests at issue.

1.5 The board does not contest that by carrying out slight variations on the process conditions described in detail in Examples 1 and 2 of the patent in suit, it might well be possible for the skilled practitioner to arrive at the preparation of some amorphous silicas falling within the ambit of the claims at issue.

1.6 However, bearing in mind that the contested patent had been revoked in particular because of the absence of details as regards the stirring speed used during the preparation of the silica claimed, the discussion at the oral proceedings focused on this crucial point whereby the appellant confirmed that stirring was an important feature and it pointed in this respect to paragraph [0049] of the patent in suit, reading:

"Mixing is an important feature in the reaction of silicate and sulphuric acid. Consequently fixed specifications, as listed in Chemineer Inc. Chem. Eng. 26 April 1976 pages 102-110, have been used to design the baffled, heated stirred reaction vessel. Whilst the turbine design is optional to the mixing geometry, a 6-bladed 30° pitched bladed unit has been chosen for the experiments in order to ensure maximum mixing effectiveness with minimum shear."

1.7 The appellant argued that the skilled person reading said paragraph would use the above suggested turbine design and owing to the teaching that "minimum shear" with "maximum mixing effectiveness" was to be ensured, he would find the information necessary to ensure the "minimum shear" in the document cited in the above passage, i.e. D10 in the present decision. It quoted in

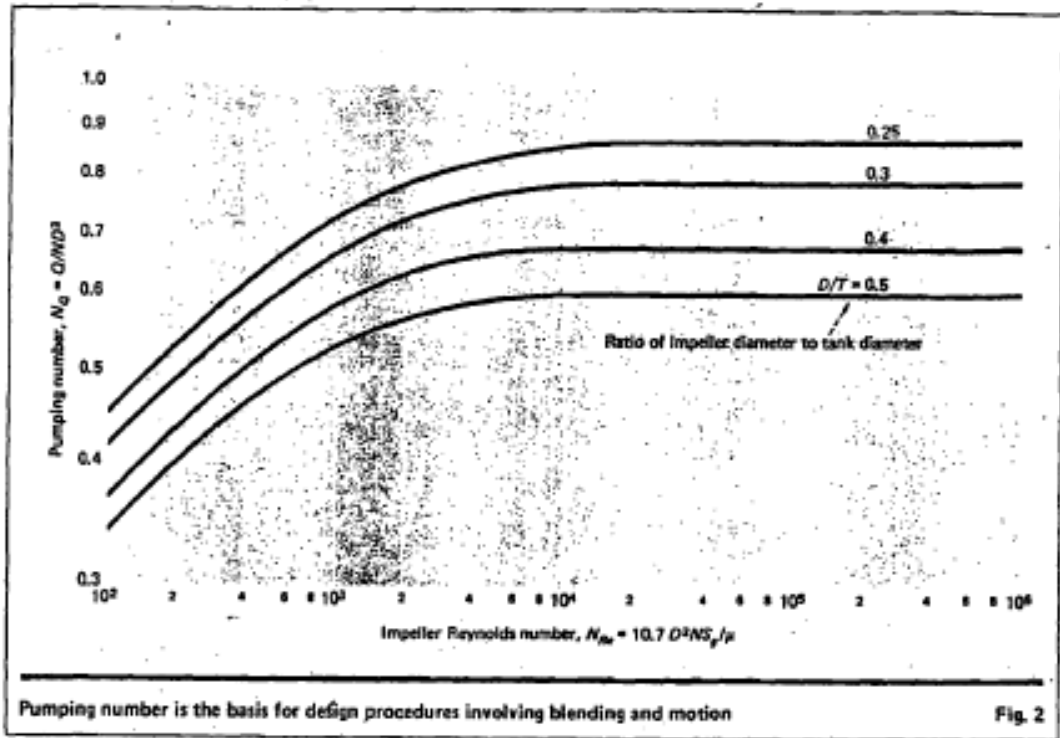
this respect Table 1 (page 103) and Figure 2 (page 104) of D10 (both quotations reproduced hereinafter).

Table 1: Process requirements set degree of agitation for blending and motion

Scale of agitation	Bulk fluid velocity, ft/min	Description
1	6	Agitation levels 1 and 2 are characteristic of applications requiring minimum fluid velocities to achieve the process result.
2	12	Agitators capable of level 2 will: - Blend miscible fluids to uniformity if specific-gravity differences are less than 0.1. - Blend miscible fluids to uniformity if the viscosity of the most viscous is less than 100 times that of the other. - Establish complete fluid-batch control. - Produce a flat, but moving, fluid-batch surface.
3	18	Agitation levels 3 to 6 are characteristic of fluid velocities in most chemical process industries agitated batches.
4	24	Agitators capable of level 6 will: - blend miscible fluids to uniformity if specific-gravity differences are less than 0.6. - Blend miscible fluids to uniformity if the viscosity of the most viscous is less than 10,000 times that of the other.
5	30	- Suspend trace solids (<2%) with settling rates of 2 to 4 ft/min.
6	36	- Produce surface rippling at lower viscosities.

7	42	Agitation levels 7 to 10 are characteristic of applications requiring high fluid velocity for the process result, such as in critical reactors.
8	48	Agitators capable of level 10 will: - Blend miscible fluids to uniformity if specific-gravity differences are less than 1.0. - Blend miscible fluids to uniformity if the viscosity of the most viscous is less than 100,000 times that of the other.
9	54	- Suspend trace solids (<2%) with settling rates of 4 to 6 ft/min.
10	60	- Provide surging surfaces at low viscosities.

Figure 2:



Relying on the content of document D10 and on the above quotations, the board does not accept this argument because neither Table 1, nor Figure 2, nor the four

excerpts submitted as D10 address the preparation of an amorphous silica, let alone the preparation of a silica having the properties presently claimed. Furthermore, neither Table 1, nor Figure 2, nor the remaining parts of D10 appear to provide the information necessary to ensure the "minimum shear" with "maximum mixing effectiveness" referred in the paragraph [0049] of the patent in suit. The appellant was invited to comment on this issue at the oral proceedings but was in particular not able to explain how the skilled person using the information found in document D10 should operate to arrive without undue experimentation at the product claimed.

Since the stirring conditions are missing as regards the preparation of the two examples, there are no concrete data on which the skilled person could rely. This means that as regards the preparation of amorphous silicas claimed other than those specifically exemplified in the patent in suit, the stirring conditions have in any case to be discovered by trial and error.

- 1.8 The appellant argued in this respect that the determination of the optimal stirring speed in the preparation of the silica claimed would be arrived at without undue burden simply by varying the stirring speed during the reaction of silicate with sulphuric acid while reworking the two examples of the patent specification.

The board can accept that such a trial and error experimentation might in the present case not be considered as undue burden as far as the silicas

illustrated in the examples of the contested patent are concerned. However, this reasoning which can be accepted only for the two examples, does not hold good for the other claimed but non-exemplified amorphous silicas and in the absence of any specific recipe concerning the preparation of such silicas, the problems concerning the stirring speed still remain for silicas claimed over the whole range.

- 1.9 The skilled person is thus confronted with the uncontested fact that he has a lot of process variables affecting the claimed parameters, but once he has encountered failure in one parameter value, there is no clear guidance enabling him to adjust the multitude of process steps in order to arrive with certitude at silicas meeting the parameter requirements defined in claim 1 of both requests at issue.

Even though a reasonable amount of trial and error is permissible when it comes to assessing sufficiency of disclosure, there must still be adequate instructions in the specification, or on the basis of common general knowledge, leading the skilled person necessarily and directly towards success, through evaluation of initial failures. This is not the case here, since the preparation of the amorphous silicas claimed is made dependent on the adjustment of different process parameters for which no guidance is given in the patent in suit, so that the broad definition of an amorphous silica as presently claimed is no more than an invitation to perform a research program in order to find a suitable way of preparing the amorphous silicas over the whole area claimed.

1.10 It follows from the above, that the principle underlying Article 83 EPC that the skilled person should be given sufficient guidance for performing the invention without undue burden over the whole range claimed is thus not fulfilled and therefore, the subject-matter of claims 1 of both requests at issue is found not to meet the requirements of Article 100 (b) EPC.

Order

For these reasons it is decided that:

The appeal is dismissed

The Registrar:

The Chairman:

C. Vodz

G. Raths