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DECISION of 29 September 1999

Case Number:

T 0586/94 - 3.3.6

Application Number:

85114009.5

Publication Number:

0185182

IPC:

C11B 3/10

Language of the proceedings: EN

Title of invention:

Method for refining glyceride oils using amorphous silica

Patentee:

W.R. Grace & Co.-Conn.

Opponent:

Laporte Industries Limited SÜD-CHEMIE AG

Headword:

Relevant legal provisions:

EPC Art. 83, 54, 56

Keyword:

"Sufficiency of disclosure (yes)"

"Novelty (yes)"

"Inventive step (yes)"

Decisions cited:

T 0228/90

Catchword:



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Boards of Appeal

Chambres de recours

Case Number: T 0586/94 - 3.3.6

DECISION of the Technical Board of Appeal 3.3.6 of 29 September 1999

Appellant: (Opponent) Laporte Industries Limited

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Decision under appeal:

Decision of the Opposition Division of the European Patent Office posted 19 May 1994

rejecting the opposition filed against European patent No. 0 185 182 pursuant to Article 102(2)

EPC.

Composition of the Board:

Chairman:

P. Krasa

Members:

H. H. R. Fessel

M. B. Günzel

Summary of Facts and Submissions

The only independent Claim 1 of European patent No. 0 185 182 reads as follows:

"Use of amorphous silica having an effective average pore diameter of greater than 6 nm (60A°) in which the average pore diameter (APD) is calculated from the measured pore volume and surface area using the equation

$$APD(nm) = \frac{4,000 \times PV(cm^3/g)}{SA(m^2/g)}$$

for the removal of phospholipids and metal ions from glyceride oils having a phosphorus content of about 230 to about 1 ppm in the absence of any solvent and without the addition of oxygen/phosphoric acid at temperatures at which the respective oils are liquid and at a concentration of the amorphous silica (calculated on a dry weight basis after ignition at 954°C) of 0.01 to 1.0% by weight, based on the weight of the oil processed."

Claims 2 to 12 relate to preferred embodiments of the subject-matter claimed in Claim 1.

II. Notices of Opposition were filed on 20 and on 22 October 1992, respectively, by Laporte Industries Limited (appellant 01) and Süd-Chemie AG (appellant 02). In their Statement of Grounds of Opposition the Opponents alleged lack of novelty, of inventive step and of sufficiency of disclosure (Article 100(a) and (b) EPC).

These objections were based inter alia on the following documents:

D1: Gutfinger et al., J.Am.Oil Chem.Soc. 1978, 55, 856-859;

D6(a): Chem.Abstr. 87:86655y,

(b): A.A. Schmidt et al., Use of synthetic sorbents for extraction of by-products from cottonseed oil, Maslo-Zhir., Prom-st., 1977(7) 21-23, Russian original of (a),

(c): English translation of (b) submitted by the Patentee;

D10: GB-A-1 255 370;

Dll: A report presented on the World Conference on Processing Palm, Palm Kernel & Coconut Oils, 11 to 16 November 1984, Kuala Lumpur, Malaysia;

D14: R. Fahn, Fette, Seifen, Anstrichmittel, 1973, 75, 77-82;

D15: FR-A-2 241 613;

D17: GB-A-228 889, and

D18: US-A-2 176 851.

III. With a decision delivered on 19 May 1994 the opposition division rejected the oppositions and maintained the patent as granted. It found the requirements of Article 83 EPC to be met and the claimed subject-matter to be novel vis-à-vis Dll. It held Dl to represent the closest prior art and that the problem to be solved was to provide a further improvement of the performance of

the silica component in the oil refining process of the prior art. It held that this problem was solved by using 0.01 to 1.0% by weight of an amorphous silica having an APD of greater than 6 nm for the removal of phospholipids and metal ions from glyceride oil having a phosphorous content of about 230 to about 1 ppm. The Opposition Division was of the opinion that neither D1 itself nor a combination with D6 or the documents D17 and D18 could lead a skilled person to the claimed solution.

IV. Appeals were filed on 15 and 27 July 1994, respectively, by the appellants. In the grounds of appeal received on 15 and 29 September 1994 the appellants disputed that there was sufficiency of disclosure inter alia with regard to

> D19: Ullmanns Enzyklopädie der technischen Chemie, Band 21 (1982), page 459, first column, paragraph 1,

a document already cited in opposition proceedings.

They also disputed novelty and argued that the claimed subject-matter had already been disclosed in D18 (appellant 02), as supported by experimental data, in D1, and in D11 disclosing e.g. acid treated montmorillonite which contained a substantial portion of silica of pores greater than 6 nm.

Moreover the appellants disputed that the alleged invention involved an inventive step and argued that in the light of

D20: Membrane Mimetic Chemistry (1982), page 127,

1801.D

also a document already cited in opposition proceedings, as well as in the light of D15 it would have been obvious to use an APD greater than 6 nm in a process as defined in Claim 1 of the patent in suit.

- V. In response to a communication from the Rapporteur the respondent (patentee) filed in addition to his main request to dismiss the appeal various auxiliary requests.
- VI. In the written proceedings and during oral proceedings held on 29 September 1999, which the appellant Laporte plc. did not attend as announced by letter dated 6 July 1999, the appellants maintained their objection as to Article 83 EPC arguing that in the absence of specifying which of the possibilities to determine the APD, which could be measured by 3 different methods, no reliable and well defined values could be obtained by the BET method for the surface area of hydrogels with 96% water content.

During oral proceedings Süd-Chemie acknowledged novelty of the claimed subject-matter over D1 and D6 but maintained the lack of novelty objection on the basis of Ullmann, 4. Auflage (1976), Band 11, pages 483 to 484 (D30) and Ullmann, 4. Auflage (1983), Band 23, pages 322 to 324 (D14a). The novelty objection raised by Laporte plc. in writing was based on D1, D10, D11, D14 and D18.

The objection as to lack of inventive step was maintained by the appellants.

VII. The respondent produced arguments as to sufficiency, novelty and inventive step.

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VIII. The appellants requested that the decision under appeal be set aside and the European patent No. 0 185 182 be revoked.

The respondent requested that the appeal be dismissed. As auxiliary requests the respondent requested that the patent be maintained on the basis of any of auxiliary requests I to V filed with the respondent's letter dated 30 August 1999, taken in their consecutive order.

Reasons for the Decision

- 1. The appeal is admissible.
- 2. Sufficiency

Appellant 02 disputed that the method to determine the APD was indicated in the patent in suit in a manner sufficiently clear and complete to be carried out by a skilled person. The Board, however, considers that the indications contained in the patent in suit, in particular the disclosed different types of test methods and the information thereupon were sufficient to enable a skilled person to select a suitable amorphous silica at the filing date of the patent in suit. The patent specification provides in detail how the required data regarding PV and SA should be determined for each type of silica (see page 5, lines 1 to 41). The Board, however, considers the claimed value of 6 nm, which is given as the lower limit of the effective APD, not to be an absolute but a relative value depending on the method to be used. There is no evidence available to the Board that amorphous silica do not exist which have an APD of greater than 6 nm at least according to one of the methods available to the skilled person. Having this in mind the Board is

satisfied that the provisions of Article 83 EPC are met, even if temperature and time of the measurement have not been specified in the patent in suit as alleged by the appellant.

Novelty

Appellant 02

In oral proceedings appellant 02 did no longer dispute novelty on the basis of D1 and D6. He, however, alleged lack of novelty with regard to D30 read in the light of D14a. Nowhere, however, in these documents the use of an amount of 0.01 to 0.1 % by weight of amorphous silica, as claimed in Claim 1, was specified. Therefore D30 does not disclose the subject-matter of Claim 1 of the patent in suit.

Appellant 01

The Opposition Division decided that novelty of the claimed subject-matter over Dll could be acknowledged on the basis of at least the essential difference that pore structure and pore diameters in Dll were attributed to the acid treated bleaching earth and not to any amorphous silica used according to Claim 1 of the patent in suit defined on page 4, lines 10 to 24 of the patent specification.

Appellant 01 alleged that the silica gel formed in Dll was an amorphous one and that there was no limitation in the patent in suit that the amorphous silica used could not be chemically bound to or within the structure of acid activated clay. Since Claim 1 of the patent in suit had to be interpreted in this broad sense its subject-matter was anticipated by Dll.

However, as the opposition division correctly stated, amorphous silica containing pores of an average diameter greater than 6 nm has not been disclosed in Dll: This document admittedly discloses that silica gel is formed at the edges of the clay particles in the course of the acid treatment of montmorillonite and discusses its influence on the surface area of the acid-treated clay. It is silent, however, on the APD of the silica gel portion of the acid treated montmorillonite in which no major structural collapse occurs (fourth page, second paragraph in the left hand column, and second page, the fourth paragraph from the bottom of the left hand column). Therefore, all information on the APD (see e.g. Figure 6) reads always on acid-activated montmorillonite in which the crystalline structure of the clay is essentially maintained. It follows that Dll does not directly and unambiguously disclose amorphous silica with an APD of greater than 6 nm.

- 3.3 No evidence was provided that the adsorbent TONSIL ACC of D1 is an amorphous silica having the characteristics specified in Claim 1 of the patent in suit. This objection was merely based on the contention that TONSIL ACC was an acid treated clay containing amorphous silica and that said silica comprised a substantial fraction having pores greater than 6 nm and that said fraction was present in an amount which would fall in the claimed range of 0.01% to 1.0% by weight. In the absence of any supporting evidence, this submission has to be dismissed as a mere allegation.
- 3.4 D18 discloses a solid silicic-acid foam to be used as an adsorbent as, for instance, in removing phosphatides and other impurities from vegetable oils (page 1, left hand column, lines 1 to 10). Neither the average pore diameter of the adsorbent nor process parameters of the purification process are disclosed in D18. Appellant 02

reworked the example of D18 and submitted that the pore diameter of the resulting product was greater than 6 nm. However, as the other features of the use according to Claim 1 of the patent in suit are not disclosed in D18, this use has not been made available to the public by D18.

Based on D10, page 1, lines 31 to 47 and D14, the paragraph headed "Ergebnisse" on page 78, appellant 01 argued that it was known to generate amorphous silica by acid treatment of clay minerals (montmorillonite, bentonite), here called "liberated silica" or "silica gel". This does not go beyond the disclosure of D11 and for the same reasons cannot be accepted as a disclosure of amorphous silica containing pores of an average diameter greater than 6 nm.

Conclusion

- 3.6 As, therefore, none of the citations D1, D6, D10, D11, D14, D18 and D30 discloses the subject-matter of Claim 1 of the patent in suit, the Board concludes that this subject-matter was not made available to the public before the priority date of the patent in suit and, consequently, is novel.
- 4. Problem and solution
- 4.1 The patent in suit relates to a method for refining glyceride oils using amorphous silica (page 2, lines 3 to 7).
- 4.2 D1 relates to the evaluation of efficiency of various adsorbents in removing phospholipids and pigments from degummed soybean oil (abstract).
- 4.3 D6 concerns the use of synthetic sorbents for extraction of byproducts from cottonseed oil.

.../...

- D18 relates to the use of a solid silicic-acid foam as 4.4 adsorbent and drying agent and to a process of producing it. This citation was suggested by appellant 02 as starting point for evaluating inventive step. This document was filed in 1937 and is directed to adsorbing agents for removing various kinds of substances from liquids containing them, as for instance, for removing phosphatides, mucilages, sterols, pigments, and associated substances from vegetable oils containing them (loc.cit., page 1, lines 5 to 10). The patent in suit (filed in 1985) addresses the refining (removing trace contaminants) of glyceride oils encompassing both vegetable and animal oils, i.e. the so-called edible oils chiefly used in foodstuffs (see page 1, lines 1 to 10). Foodstuffs, however, had to meet far more strict specifications in 1985 than in 1937 with the consequence that a teaching encompassing, as mentioned above, under various kinds of substances to be removed from liquids as an example also the removal of phosphatides from vegetable oils cannot be considered to hint at the use of the amorphous silica of the patent in suit with the expectation that by the use of these adsorbents the resulting edible oils will meet the high standards required about 50 years later. Moreover, D18 contains no quantitative data at all on the purification effects to be achieved by the application of the disclosed adsorbent. Under these circumstances, the Board finds it unrealistic to assume that a skilled person would have tried to improve the art disclosed in D18. Therefore, the Board decides that D18 does not qualify as a starting point for defining the technical problem.
- 4.5 The Board takes D1 as starting point for evaluating inventive step; this citation was considered by the opposition division as representing the most relevant prior art.

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The problem to be solved versus this prior art can be seen in a further improvement of the performance of the silica component in the oil refining process as defined in Claim 1 in particular in a (further) reduction of the oil's phosphorus and metal contents.

- 4.6 In view of the results given in Table V and Table VI of the patent in suit the Board is satisfied that the claimed subject-matter solves the existing technical problem as defined above.
- 5. Inventive step

It has now to be considered whether the means claimed in Claim 1 involve an inventive step.

5.1 D1 teaches in Table I the use of different adsorbents for the removal of phospholipids from soybean oil (a glyceride oil) having (i) a phosphorus content of 17.3 μ g/g oil, (ii) in the absence of any solvent, (iii) without the addition of oxygen/phosphoric acid, (iv) at a temperature at which the oil is liquid (90-120°C), and (v) at a concentration of 1 to 5 % w/w.

Table I of D1 demonstrates in particular the effect of different adsorbents on phospholipid content and colour. It may be seen therefrom that activated clays (Tonsil clays) have higher adsorbing capacities than natural Earth (Fuller's Earth) for both colour bodies and phospholipids. Moreover it is stressed that the behavior of the activated clays (Tonsil L80 and Tonsil ACC) is not the same when the adsorbables are phospholipids or colour bodies (cf. Figures 1 and 2 on page 858 and last complete paragraph on the same page). As to the other adsorbents listed in that table, it may be seen that the bleaching power of silicic acid and Florosil is very low and much worse than their ability to adsorb phospholipids (cf. page 858). It is also

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demonstrated that the ability of silicic acid to adsorb phospholipids is low compared with that of Tonsil. These experimental findings demonstrate that the adsorbing capacities vary in an unpredictable manner not only with the adsorbent used but also with the respective adsorbables.

D1 does thus not provide any hint that the above problem may be solved by amorphous silica. To the contrary, it is shown that the adsorption capacity of specific clays would be much better than that of silicic acid as shown in the Table for 1 weight % of Tonsil 80 compared with 2 weight % silicic acid.

Moreover D1 is silent as to the removal of metal ions being part of the problem to be solved.

- The Board considers that this teaching remains unchanged even if considering D30 dealing in point 1.7.3.1. with decolourization of oils by adsorbents. In that document it is taught in very general terms that said adsorption aims not only at decolourization but at the same time at the removal of soaps, phosphatides, traces of metals and oxidation products. Activated bleaching earth is said to be an excellent adsorbent in amounts of 0.5 to 1% (page 484, left column, lines 10 to 12). This adds nothing to the teaching already known from D1.
- 5.3 From D6 a skilled person would not get any incentive to depart from applying Tonsil clays which are the most effective adsorbents according to D1 since D6(b) refers to "miscellas" and thus clearly describes a solvent based refinement process which a skilled person would not consider when dealing with a solvent free process. Moreover, from Table I of D6(b) it can be seen that the relation of oil to silica ranges from 2:1 to 1:2, which is intolerable for any economical process. Therefore the results of D6(b), if considered at all, could not

lead a skilled person to use such material in a solvent free process, attempting to remove phospholipids and metal ions from glyceride oils with the intention of reducing the amount of adsorbent needed.

The Board accepts the appellants'argumentation that D6(a), the abstract of D6(b), discloses that for the sorption of coloured impurities and phospholipids from cottonseed oil by silica gels, the optimum pore size is in the range of 6 to 8 nm; but the Board does not accept the argumentation that the disclosure encompasses the use of these sorbents in a solvent free process.

It is agreed that said abstract is silent as to the use of a solvent in the oil refining process. However, such silence on the presence or absence of a solvent in the adsorption process cannot be construed as meaning "absence of a solvent" but can only be taken as what it is: lack of information on this feature. In such a situation, a skilled person who knows that an abstract contains the information of the original document in an abridged and often incomplete form will have recourse to the original - provided it is available - to fill that gap of information (see T 0228/90, reasons for the decision point 5.1.2; not published in the OJ EPO). In the present case, the original D6(b) relates to the use of synthetic sorbents for separating accompanying substances from "miscellas" which means to a solvent based refinement of cottonseed oil as may be seen from its translation into English, D6(c).

A skilled person would not expect the absorptions characteristics of an adsorbent to be the same with and without solvent. It follows that a skilled person would not have found any incentive in D6 to try a solvent-free oil refining process when looking for a solution of the existing technical problem.

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- For the same reasons, a skilled person would not have profited from the technical information revealed in D15 when looking for a solution of the existing technical problem. This citation also relates to a refinement process for improving the storage properties of edible oils (page 1, lines 1 to 7). In this process the presence of a solvent is mandatory (see page 2, lines 35 to 37), contrary to the process of Claim 1 of the patent in suit.
- D20 is a theoretical paper dealing with phospholipids 5.5 and the formation of phospholipid aggregates via hydrogen bonding in the presence of water. Appellants contended that when seeking to adsorb phospholipids the skilled person would have taken into account the dimensions of these aggregates and would have concluded that an APD of greater than 6 nm was required. They argued that, therefore, the use of adsorbents with such an APD for the removal of phospholipids had been obvious. The Board cannot accept this conclusion since D20 contains no information whether or not these phospholipid aggregates would still exist under the conditions of the present adsorption process and whether or not their dimensions would have any bearing on the latter.
- 5.6 For these reasons, the Board finds that the subjectmatter of Claim 1 of the patent in suit was not
 rendered obvious by the citations D1, D6, D15, D18,
 D20, and D30, either alone or in combination. It
 involves, therefore, an inventive step. Dependent
 Claims 2 to 12 relate to particular embodiments of the
 subject-matter of Claim 1 and derive their
 patentability from the latter.

Under these circumstances it was not necessary to deal with the respondent's auxiliary requests.

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Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

G. Rauh

P. Krasa