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Datasheet for the decision of 23 July 2021

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Application Number:	09848620.2
Publication Number:	2471743
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Language of the proceedings:	EN

Title of invention:
MESOPOROUS COMPOSITE TITANIA AND PREPARING METHOD THEREOF

Patent Proprietor:
Nanjing University of Technology
Nanjing Taiwei Technology Co., Ltd.

Opponent:
Huntsman P&A Germany GmbH

Headword:
Mesoporous titania/Nanjing University

Relevant legal provisions:
EPC Art. 100(b), 100(a)

Keyword:
Sufficiency of disclosure - (yes)
Novelty - (yes)
Inventive step - (yes)

Decisions cited:
T 0464/94, T 0593/09, T 0045/10

Catchword:

Europäisches Patentamt
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Beschwerdekammern

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Case Number: T 1326/19 - 3.3.05

D E C I S I O N
of Technical Board of Appeal 3.3.05
of 23 July 2021

Appellant: (Opponent)	Huntsman P&A Germany GmbH Dr.-Rudolf-Sachtleben-Strasse 4 47198 Duisburg (DE)
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Representative:	Kramer Barske Schmidtchen Patentanwälte PartG mbB European Patent Attorneys Landsberger Strasse 300 80687 München (DE)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted on 1 April 2019 rejecting the opposition filed against European patent No. 2471743 pursuant to Article 101(2) EPC.**

Composition of the Board:

Chairman	E. Bendl
Members:	G. Glod
	P. Guntz

Summary of Facts and Submissions

I. The opponent's (appellant's) appeal lies from the opposition division's decision rejecting the opposition against European patent No. 2 471 743 B1.

Claim 1 of the patent is as follows:

"*A mesoporous composite titanium oxide composed of a mesoporous titanium oxide and an inorganic matter composited on the outside surface and the wall of pores of the mesoporous titanium oxide; said inorganic matter contains at least one element selected from sulphur, phosphorus and selenium in an amount of 0.01 %-25 %, based on the amount of the element mass, of the mass of said mesoporous composite titanium oxide material; wherein at least one most probable pore diameter of pore distribution of the mesoporous composite titanium oxide material is 3-15 nm, the specific surface area is 50-250* m^2/g*, and the pore volume is 0.05-0.4* cm^3/g*.*"

Claims 2 to 10 directly or indirectly relate to claim 1.

II. The following documents are of relevance here:

D1: WO 2009/018700 A1
D1a: EP 2 189 420 A1, family member of D1, referred to herein when making reference to D1
D3: EP 1 092 472 A1
D4: DE 100 57 105 A1
D16: CN 1 850 333 A incl. translation of the Claims
D17: DE 102 32 868 A1
D18: Sachtleben Pigment GmbH, Certificate of Analysis, 19 January 2009, Lot UBF001400

D19: Hombikat AK1-Lot UBF1400
D20: Huang et al.; China Particuology, Vol. 3, No.3, 2005, pages 176-180
D21: Li et al., Journal of Molecular Catalysis A: Chemical 226, 2005, pages 93-100
D22: JP 2007-66668 A and translation (Patent Translate)
D23: US 2006/0210798 A1
D24: US 2004/0238410 A1
D27: Xekoukoulotakis, N.P. et al.; Proceedings of the 10th International Conference on Environmental Science and Technology; Kos Island, 5-7 September 2007.

III. The appellant's arguments relevant to the present decision can be summarised as follows:

Article 100(b) EPC was prejudicial to the maintenance of the patent. The methods allowing the parameters of claim 1 to be measured were not present in claim 1 or in the description. The expression "at least one most probable pore diameter" was not understood by the skilled person. Even reference [3] of the patent in suit did not provide enough information to correct this deficiency.

The subject-matter of claim 1 lacked novelty in view of each of D1, D4, D16, D17, D20, D24 and the prior use as illustrated by D18, D19 and D27.

In particular, the process according to D1 for preparing the mesoporous titania included a washing step that was possibly carried out with a sulphuric acid solution. The lower level of the amount of inorganic matter of 0.01 to 25% was inevitably exceeded by the material known from D1. It could be assumed that

the washing step with glycerol did not remove all sulphate ions from the surface of the D1 material.

It was clear from D4 that sulphate was sufficiently present in the range given in claim 1 of the patent in view of the fact that titanyl sulphate was used as the starting material. This was also confirmed by the indications in examples 1 and 2 that SO_4^{2-}-ions were present on the wall of pores.

In view of the given surface area and the mesoporous properties of the material of D16, it was derivable by calculation that the pore volume had to be in the range of claim 1 of the patent.

Material A of Table 1 of D17 anticipated the novelty of claim 1 of the patent.

The public availability and sale of Hombikat AK1, as documented in D18, D19 and D27, made all its features available to the public.

It was evident that the calcined TiO_2 materials, shown in Table 2 of D20, using phosphoric acid as a catalyst, had a phosphorus content in the range given in claim 1, since drying did not allow complete removal of phosphoric acid.

The subject-matter of claim 1 also lacked an inventive step, when starting from D1 as closest prior art. Embodiment 2 of Table 1 of the patent was representative of D1. The problem to be solved could only be seen in the provision of an alternative. The solution was obvious since, starting from embodiment 5 of Table 1, the skilled person would have replaced

formic acid by sulphuric acid and obtained a titanium oxide as claimed.

IV. The appellant (opponent) requested that the decision under appeal be set aside and that the European patent be revoked.

The respondent (patent proprietor) did not make any submissions as to the substance of the appeal. The implicit request was, thus, that the appeal be dismissed.

Reasons for the Decision

1. Article 100(b) EPC

This ground does not prejudice the maintenance of the patent as granted for the following reasons:

1.1 The parameters present in claim 1 are not ill-defined within the meaning of T 593/09 (Reasons 4.1.4). These parameters are known to the skilled person, who will know that several methods exist for their determination.

Paragraph [0004] of the patent discusses the starting point and background of the patent, including methods for preparation of mesoporous titanium oxide, whereby reference [3] is explicitly mentioned. This reference from "Chemical Communication", referred to in paragraph [0037], also includes, as supplementary material, the figures. It discloses textural properties of titania samples and describes how the data is obtained. In particular, it is disclosed that the porosity of the titania materials was investigated using nitrogen adsorption-desorption isotherms (right-hand column,

first sentence, second full paragraph). The skilled person reading the patent will consider this reference since it is the starting point of the claimed invention. The skilled person will therefore use the method disclosed therein and accompanying calculations to obtain the required data. The appellant has not provided any evidence that would question the measurements shown in Table 1 of the patent and prove that the methods disclosed in reference [3] are not suitable for the examples of the patent.

In Table 1 of the reference, $D_{BJH/nm}$ is indicated. It is evident that this value is not the average pore diameter, but the maximum of the pore-size distribution plots calculated by the BJH (Barrett–Joyner–Halenda) equation from the adsorption branch of the isotherm and shown as an insert to Figure S3 for different temperatures. The skilled person understands that this value is considered a most probable pore diameter within the meaning of the patent. The fact that at least one most probable pore diameter of pore distribution should be within the range claimed simply indicates that a multimodal particle size distribution is possible and that one of the maxima should be in the claimed range.

Decision T 45/10 is not relevant to the present case, since the patent underlying the decision T 45/10 did not refer to a reference describing the methods that can be used.

1.2 The objection relating to claim 3 and the details about the starting materials cannot be accepted. Paragraph [0020] provides further details about the precursors that should be used and cites thiophenol as a preferred S-component. There is no evidence that this component

cannot be used in the preparation of the desired mesoporous titanium oxide.

2. Article 100(a) EPC together with Article 54 EPC

Likewise, this ground does not prejudice the maintenance of the patent as granted for the following reasons:

2.1 D1 discloses in claim 1 a method for preparing titania with controllable microporous-mesoporous structure, wherein alkali metal titanate is used as a raw material, after reacting in a moisture atmosphere with a humidity of 2-100% at a temperature of 20-250°C for 0.5-72 hrs, washed with water or an acid solution, and finally subjected to air roasting or solvent thermal treatment, in order to obtain titania with the most probable pore size of 1-20 nm, a pore volume of 0.05-0.4 cm^3/g, and a specific surface area of >30 m^2/g. The acid solution can be a sulphuric acid solution (claim 8). Embodiment 2 explicitly discloses the preparation in the presence of sulphuric acid and a post-treatment with glycerol. Even if it were assumed that the post-treatment with glycerol did not remove all sulphate ions - for which evidence is lacking - D1 still does not disclose the concentration of the sulphate ions in or on the wall. It may be highly likely that the concentration is in the claimed range, but whether a document is prejudicial to novelty cannot be decided on the basis of probability (see T 464/94, Reasons 16).

2.2 D4 discloses, in example 2-2, titanium oxide having a specific surface area of 59 m^2/g, a pore volume of 0.09 cm^3/g and an average pore diameter of 6.1 nm. Irrespective of whether the average pore diameter can

be considered as the most probable pore diameter, D4 does not disclose the concentration of sulphate in the pores. The appellant argued that it should be clear that it was in the claimed range, but has not corroborated its statement by evidence. Therefore, a direct and unambiguous disclosure of the claimed concentration of sulphur in or on the pores is not present in D4.

2.3 The appellant has not provided any indication concerning why the opposition division's reasoning in point 2.3 of the impugned decision, in particular the silence of D16 with respect to the pore volume, is wrong.

2.4 Material A shown in Table 1 of D17 has an average pore diameter of 14.8 nm, a pore volume of 0.33 cm^3/g and a surface area of 75 m^2/g . There is no disclosure of the amount of sulphate in the final support. According to claim 3 the support made of titanium dioxide contains 1.0 to 5.5 mass-% of free sulphate. Even if it were accepted that this feature must also apply to material A of Table 1, then it is still not directly and unambiguously derivable that the average pore diameter of 14.8 nm implies a maximum in the pore size distribution in the range of 3-15 nm.

2.5 In its reasoning, the opposition division found lack of evidence of sale of the analysed product of D18, lack of a clear link between D18 and D19, and no link to D27. Finally, there was no proof that the products analysed in D18 and D19 had sulphate on the walls of the pores. The appellant has not provided any reasoning concerning why the opposition division's position was erroneous.

In view of the lack of evidence regarding the alleged prior use and the lack of data - especially evidence showing the presence of sulphur on the outside surface and the wall of pores - with respect to the allegedly sold product, there was no need to hear the proposed witness (never actually nominated) to corroborate the presence of sulphur on the outside surface and the wall of pores. Thus, since there is no proof that a possible sale of Hombikat AK1 might have disclosed all of the claimed features and could be novelty-destroying, there was no need to hear a witness on the questions of public availability and existence of other features.

2.6 Table 2 of D20 shows mesoporous titanium oxides obtained with phosphoric acid and dried either by vacuum or IR and having, respectively, a surface area of 63 and 101 m^2/g, an average pore size of 7.1 and 6.0 nm, a total pore volume of 0.113 and 0.152 cm^3/g. Furthermore it is indicated that phosphoric acid cannot be removed completely through drying. However, the amount of phosphorus not removed by drying is not disclosed. As in the case of D1, it may be highly likely that the concentration is in the claimed range, but whether a document is prejudicial to novelty cannot be decided on the basis of probability (see T 464/94, Reasons 16).

2.7 No reasoning is provided for D24 except for indicating paragraph [0068]. The board cannot find the features of claim 1 in said paragraph.

3. Article 100(a) EPC together with Article 56 EPC

Likewise, this ground does not prejudice the maintenance of the patent as granted for the following reasons:

3.1 The invention relates to a mesoporous composite titanium oxide.

3.2 D1 is considered the closest prior art since it relates to the preparation of titanium oxide with microporous-mesoporous structure. It also addresses the use of such compounds in lithium-ion batteries (paragraph [0003]).

As indicated above (point 2.1), D1 discloses in claim 1 a method for preparing a titanium oxide having porosity ranges that overlap with those of claim 1 of the patent. The washing solution could be sulphuric acid and the final treatment could be with a solvent. There is thus no direct and unambiguous disclosure in the general part of the description and claims of a titanium oxide having inorganic matter composited on the outside surface and the wall of pores.

There is no evidence that embodiment 5 discloses a mesoporous titanium oxide having carbon composited on the outside surface and the wall of pores in an amount of 0.01%-25%. It does not appear to be implicit that the post-treatment with ethylene glycol will inevitably lead to the deposition of carbon, since process details of the post-treatment are lacking.

Still to the appellant's advantage it is accepted that embodiment 5 of D1 is in line with embodiment 2 of the patent.

3.3 Based on this consideration, the problem to be solved is to provide an alternative mesoporous titanium oxide.

3.4 The problem is solved by a mesoporous composite titanium oxide according to claim 1, characterised in

that it contains an inorganic matter composited on the outside surface and the wall of pores of the mesoporous titanium oxide, and in that said inorganic matter contains at least one element selected from sulphur, phosphorus and selenium.

3.5 The proposed solution does not appear obvious for the following reasons:

D1a discloses that several washing solutions including formic acid and sulphuric acid can be used (claim 8). Even if the skilled person replaced formic acid by sulphuric acid in embodiment 5, there is still no evidence that this would lead to the presence of sulphur on the outside surface and the wall of pores of the mesoporous titanium oxide in the claimed concentration.

D4 relates to a different preparation process, and the skilled person would not therefore turn to D4. Even if considering the material of D4 as a possible alternative, the skilled person would not arrive at the subject-matter claimed (see point 2.2).

This also applies to D17, which discloses a completely different preparation process (paragraph [0090]).

D20 discloses the influence of four different inorganic acids on the structure of mesoporous titanium oxide materials without focusing on their presence in the final product. It certainly does not teach adding sulphur or phosphorus to a mesoporous titanium.

In fact, none of D3, D21, D22, D23, D25 or D26 provides such teaching to obtain sulphur, phosphorus or selenium on the outside surface and the wall of pores in the

claimed amount, while maintaining the porosity characteristics of embodiment 5 of D1.

3.6 The subject-matter of claim 1 involves an inventive step. The same applies to the subject-matter of claims 2 to 10, which directly or indirectly relate to claim 1.

Order

For these reasons it is decided that:

The appeal is dismissed

The Registrar: The Chairman:

C. Vodz E. Bendl

Decision electronically authenticated