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Datasheet for the decision of 3 April 2009

T 0829/06 - 3.3.06 Case Number:

Application Number: 99906269.8

Publication Number: 1060218

IPC: C09C 1/02

Language of the proceedings: EN

Title of invention:

Method for improving the stability of a slurry

Patentee:

Kemira Oyj

Opponent:

Solvay (Société Anonyme)

Headword:

Slurry stabilization/KEMIRA

Relevant legal provisions:

Relevant legal provisions (EPC 1973):

EPC Art. 56

Keyword:

"Inventive step (all requests) - no: obvious modification"

Decisions cited:

Catchword:



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Boards of Appeal

Chambres de recours

Case Number: T 0829/06 - 3.3.06

DECISION
of the Technical Board of Appeal 3.3.06
of 3 April 2009

Appellant: Kemira Oyj

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Decision under appeal: Decision of the Opposition Division of the

European Patent Office posted 5 April 2006 revoking European patent No. 1060218 pursuant

to Article 102(1) EPC 1973.

Composition of the Board:

Chairman: P.-P. Bracke
Members: P. Ammendola

U. Tronser

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Summary of Facts and Submissions

- I. This appeal is from the decision of the Opposition Division to revoke the European patent No. 1 060 218 concerning a method for improving the stability of slurries.
- II. The Opponent had sought revocation of the patent in suit on the grounds of lack of novelty and inventive step. It referred, inter alia, to document
 - (5) = US-A-5 658 467.
- III. During the opposition proceedings the Patent Proprietor filed with letter dated 31 May 2004 the document
 - (7) = test report

as well as a set of amended claims labelled as main request.

Claim 1 of the main request read:

- "1. A method for improving the storability of an aqueous slurry which contains inorganic solids against microbial growth, characterized in that there is added to the slurry a peracid in an amount of 200-5000 g/metric ton of dry solids, calculated as a 100 per cent peracid, said peracid being peracetic acid, persulfuric acid, perdisulfuric acid or performic acid."
- IV. In its decision the Opposition Division considered, inter alia, that the method defined in this claim lacked

novelty and that the claimed uses according to the then pending auxiliary requests were obvious for the skilled person starting from the synergistic mixtures of peracid and conventional non-oxidizing biocides disclosed in example 19 of document (5).

V. The Patent proprietor (hereinafter Appellant) lodged an appeal against this decision.

It filed with letter dated 3 March 2009, inter alia, sets of amended claims respectively labelled as auxiliary requests 1 to 13.

VI. Claim 1 of auxiliary request 1 only differs from that of the main request (see section III of the Facts and Submissions above) in that the final wording "performic acid." has been replaced by "performic acid, wherein the aqueous slurry which contains inorganic solids is a waste pigment slurry.".

Claim 1 of auxiliary request 2 only differs from that of the main request in that the wording ", persulfuric acid, perdisulfuric acid" has been deleted.

Claim 1 of auxiliary request 3 only differs from that of auxiliary request 1 in that the wording "waste" has been deleted.

Claim 1 of auxiliary request 4 only differs from that of the main request in that the range "200-5000" has been replaced by "200-4000".

Claim 1 of auxiliary request 5 only differs from that of auxiliary request 4 in that the wording ", persulfuric

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acid, perdisulfuric acid or performic acid" has been deleted.

Claim 1 of auxiliary request 6 only differs from that of auxiliary request 3 request in that the final wording "pigment slurry." has been replaced by "pigment slurry, wherein the pigment is gypsum.".

Claim 1 of auxiliary request 7 defines the use corresponding to the method of claim 1 of the main request; it reads:

"1. Use of a peracid in an amount of 200-5000 g/metric ton of dry solids, calculated as a 100 per cent peracid, for improving the storability of an aqueous slurry which contains inorganic solids against microbial growth, wherein said peracid is added to the slurry, and wherein said peracid is peracetic acid, persulfuric acid, perdisulfuric acid or performic acid."

Claim 1 of auxiliary request 8 defines the use corresponding to the method of claim 1 of auxiliary request 1; it only differs from that of auxiliary request 7 in that the final wording "performic acid." has been replaced by "performic acid, wherein the aqueous slurry which contains inorganic solids is a waste pigment slurry.".

Claim 1 of auxiliary request 9 defines the use corresponding to the method of claim 1 of auxiliary request 2; it only differs from that of auxiliary request 7 in that the wording ", persulfuric acid, perdisulfuric acid" has been deleted.

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Claim 1 of auxiliary request 10 defines the use corresponding to the method of claim 1 of auxiliary request 3; it only differs from that of auxiliary request 8 in that the wording "waste" has been deleted.

Claim 1 of auxiliary request 11 defines the use corresponding to the method of claim 1 of auxiliary request 4; it only differs from that of auxiliary request 7 in that the range "200-5000" has been replaced by "200-4000".

Claim 1 of auxiliary request 12 defines the use corresponding to the method of claim 1 of auxiliary request 5; it only differs from that of auxiliary request 11 in that the wording ", persulfuric acid, perdisulfuric acid or performic acid" has been deleted.

Claim 1 of auxiliary request 13 defines the use corresponding to the method of claim 1 of auxiliary request 6; it only differs from that of auxiliary request 10 in that the final wording "pigment slurry." has been replaced by "pigment slurry, wherein the pigment is gypsum.".

- VII. The Opponent (hereinafter Respondent) filed with a facsimile of 1 April 2009, inter alia, the document
 - (10) = Ullmann's Enciclopedia of Industrial Chemistry, Volume A 18, 1991, pages 545, 611, 612, 628 to 633, 640 and 641.

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In this facsimile the Respondent also specifically referred for the first time in these appeal proceedings to example 16 of document (5).

VIII. On 3 April 2009 oral proceedings took place before the Board as scheduled.

During the hearing the parties discussed the issue of inventive step and the Appellant filed two further sets of amended claims respectively labelled as auxiliary request 5a and 12a.

The Appellant finally announced to be also prepared to modify all its pending requests by introducing in each claim 1 a lower limit of 10% by weight for the solid content of the slurry, in order to deprive of any relevance the reference to example 16 of document (5) only mentioned by the Respondent two days before the oral proceedings.

- IX. Claim 1 of auxiliary request 5a and that of auxiliary request 12a filed during the hearing define a method and its corresponding use. They differ respectively from claim 1 of auxiliary request 6 and that of auxiliary request 13 only in that the final wording "the pigment is gypsum." has been replaced by "the pigment is gypsum or calcium carbonate.".
- X. The Appellant presented in writing and orally the following arguments in support of its opinion that the claimed subject-matter was not obvious for the skilled person starting from document (5).

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Both the methods and the uses defined in the different versions of claim 1 according to the Appellant's requests required the actual occurrence of microbial growth inhibition and implied the absence of non-oxidizing biocides, even though, in the Appellant's opinion, this latter feature was more self-evident in the use claims.

The technical problem addressed in the patent in suit was that of providing pigment slurries with a long term stability against microbial growth, while avoiding the use of the conventional non-oxidizing biocides that were considered possibly harmful to the environment.

A similar problem had been previously addressed, *inter alia*, in document (5), that, therefore, represented a reasonable starting point for the assessment of inventive step.

All versions of the method or use claim 1 according to the pending requests differed from the prior art of document (5) already because these claims required implicitly the absence of any non-oxidising biocide and explicitly a specific ratio between the amount of the peracetic acid (hereinafter PAA) and that of the solids in the slurry. On the contrary, the examples 1 to 18 of document (5) described the treatments of paper mill process waters containing an undisclosed amount of solids. Only example 19 of this citation actually disclosed the used amounts of both PAA and of the solid forming the slurry, but the resulting contents of PAA for tons of solid were lower than the 200 g/ton required in the present claims.

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Even though example 19 of document (5) was the sole actually achieving a somewhat prolonged (over four weeks) antimicrobial activity, still an even more prolonged stability had been achieved by the methods and the uses of the invention, as evident from Table 5 in the patent in suit and from the additional experimental data reported in document (7).

The Appellant conceded that, in general, to increase the initial concentration of a biocide represented an obvious measure for prolonging the time for which the antimicrobial effects of such ingredient remained detectable. However, the skilled person starting from example 19 of document (5) also derived from the reference tests given in that very example and from the whole of the disclosure in this citation that by using PAA alone no long term stability was achievable. Such citation did not disclose any amount of PAA as clearly sufficient for providing long term stability.

Moreover, the data of example 16 of document (5) apparently proving the superior biocidal activity of PAA alone in comparison with its synergistic mixture with non-oxidizing biocides, were only relevant for short term stability and, in any case, at odds with the other data reported in the same citation, as well as hardly compatible with the explicit indication in the description of the same document (5) that none of the commercially available biocides were sufficiently stable so as to exhibit a prolonged biocidal effect.

Hence, it was only with hindsight that the skilled person starting from document (5) could have modified this prior art so as to arrive at the subject-matter

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claimed in any of the main request and of auxiliary requests 1 to 5 and 7 to 12.

The non-obviousness of the claimed subject-matter would be even more evident in the case of the methods and uses according to any of auxiliary requests 5a and 6 and 12a and 13, since these latter were limited to slurries wherein the pigment were gypsum and/or calcium carbonate, i.e. solids providing, for the reasons indicated in the paragraph [0013] of the patent in suit, a longer lasting biocidal effect.

XI. The Respondent submitted in writing and orally the following arguments.

Neither the method claims nor the use claims of the Appellant's requests excluded implicitly the possible additional presence of conventional non-oxidizing biocides. Moreover, the inhibition of the microbial growth was not a technical feature of the claimed methods.

The data in the patent in suit and in the test report of document (7) suggested that the aimed long term inhibition of microbial growth had not been achieved over the whole claimed range. Moreover, neither the general description nor the examples in the patent in suit mentioned or implicitly suggested any criticality of the kind of pigment in the slurry or of the nature of the slurry, in view of the aimed long term stability or of some further technical advantage.

The PAA was explicitly acknowledged in document (5) to have been used for years at high concentrations for

inhibiting microbial growth in other kinds of slurry. The technical teaching also provided by in this citation as to the short term stability of conventional biocides did manifestly not apply to PAA. On the contrary, example 19 contained also an extrapolated value for the concentration of PAA that could only reasonably be interpreted as the amount actually required for achieving a satisfactory anti-microbial stability over several weeks.

In any case, the anti-microbial effects of PAA alone in pigment slurries were already explicitly disclosed in the reference tests of the examples of document (5).

Additionally, at least in example 16 therein the biocidal effects achieved from PAA alone were even better than those provided by the synergistic mixture.

It was therefore also self-evident to the skilled reader of document (5) that increasing the amount of PAA above the amount thereof already present in the synergistic mixture and in the reference tests disclosed in this citation would certainly allow to prolong microbial inhibition even in the absence of any non-oxidizing biocide. Therefore, the claimed methods and uses just represented an obvious alternative to the prior art.

XII. The Appellant requested that the decision of the first instance be set aside and that the patent be maintained on the basis of the set of claims according to the main request filed with letter dated 31 May 2004 or alternatively any of auxiliary requests 1 to 5 or 6 to 12 or 13 submitted with letter dated 3 March 2009 or

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auxiliary requests 5a or 12a submitted during the oral proceedings.

The Respondent requested that the appeal be dismissed.

Reasons for the decision

All requests of the Appellant

Inventive step assessment for the subject-matter of claim 1 of any of the main request and of auxiliary requests 1 to 5, 5a, 6 to 12, 12a and 13 (Article 56 EPC 1973)

The requests of the Appellant are so formulated that each claim 1 of auxiliary requests 7 to 12, 12a and 13 define the use corresponding to the method of each claim 1 of the main request and of auxiliary requests 1 to 5, 5a and 6, respectively (see above sections III, VI and IX of the Facts and Submission).

The Appellant has considered that any of these method or use claims required the occurrence of microbial growth inhibition and implicitly excluded the possible presence of non-oxidizing biocides.

It has become apparent to the Board that, even if one assumes, for the sake of argument in favour of the Appellant, that this restrictive interpretation of the claims were correct, still each claim 1 of any of the requests of the Appellant comprises subject-matter that is obvious in view of the prior art.

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In particular, even when accepting the restrictive Appellant's interpretation of the claims, the subjectmatter of each claim 1 of the main request and of auxiliary requests 1 to 5, 5a and 6, still embraces methods comprising a step in which microbial growth is inhibited in gypsum waste slurries (hereinafter GWS) because of the presence therein of exclusively PAA as the sole biocide in an amount, calculated as a 100 per cent peracid, of 200 to 4000 g/ton of dry solids. Similarly, the restrictive Appellant's interpretation of each claim 1 of auxiliary requests 7 to 12, 12a and 13 embraces the uses in GWS of exclusively PAA in an amount of 200 to 4000 g/ton of dry solids as the sole biocide for inhibiting microbial growth. Hereinafter such claimed methods certainly embraced by all method claims 1 and such claimed uses certainly embraced by all use claims 1 are cumulatively indicated as the claimed PAA/GWS methods and uses.

Hence, only the reasons for the finding of the Board that the prior art renders obvious the claimed PAA/GWS methods and uses are given hereinafter.

- 1.1 The patent in suit (see paragraphs [0004], [0010], [0013] and [0016]) mentions the following advantageous technical effects possibly relevant for the claimed PAA/GWS methods and uses:
 - a) to achieve a long term antimicrobial stability in slurries of solids such as, in particular, the waste pigment slurries produced during paper coating process,

and

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- b) to reduce the harm to environment in comparison to that produced by the conventional biocides for solid slurries.
- 1.2 It is undisputed that the synergistic mixture of PAA with non-oxidizing biocides disclosed in document (5) (see column 2, lines 1 to 31) aims at providing advantageous technical effects at least similar to those indicated in the patent in suit.

The Board notes further that document (5) discloses several examples of biocide addition to paper mill process waters of unknown solid contents (such as the "mill furnishes" of examples 1, 3 and 6, or the "whitewaters" of examples 7, 8, 10, 11 and 14 to 17, or the "service water" of examples 12 and 13). Furthermore, in all these examples the microbial growth was monitored for not more than 24 hours.

Only example 19 of this citation discloses both the amount of PAA used and the dry solid content of the slurry and, thus, allows to determine the relative amount of PAA per ton of dry solids in the slurry.

Moreover, this is the example in document (5) in which the stabilization against microbial growth has been tested for the longest time, i.e. for up to four weeks.

Therefore, the Board concurs with the Appellant that example 19 of document (5) represents a reasonable starting point for the assessment of inventive step.

1.3 The slurry of example 19 is a concentrated kaolin pigment dispersion and, thus, is not a waste pigment slurry. This example, similarly to all the others also - 13 - T 0829/06

reported in document (5), aims manifestly at demonstrating the synergistic effect achieved when using a combination of PAA with a conventional non-oxidizing biocide. To this scope it comprises also some reference tests carried out by using PAA as the sole biocide. In all the invention and reference tests of this example the amounts of PAA used per ton of solids (kaolin) are always not larger than 182 g/ton, i.e. below the lower limit of 200 g/ton as required in the claimed PAA/GWS methods and uses.

Hence, the claimed PAA/GWS methods and uses differ from the tests in example 19 wherein long term stabilization is achieved by using a synergistic mixture of PAA and non-oxidizing biocide, in:

- the larger amount of PAA used,
- the absence of any non-oxidizing biocides,

and

- the slurry is a gypsum slurry obtained from waste waters and not a freshly prepared kaolin slurry.
- 1.3.1 The Board notes that the patent in suit acknowledges this prior art in paragraph [0006] without stating or suggesting that the methods and uses of the invention could be more advantageous than those based on the synergistic mixture disclosed in document (5). Indeed, this paragraph of the patent in suit only stresses that the reference tests based exclusively on PAA also disclosed in example 19 of this citation exhibit no long lasting inhibiting effect on microbial growth.

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Hence, and in the absence of any experimental comparison with this prior art, the Board can only conclude that the level of antimicrobial stability achieved e.g. by the claimed PAA/GWS methods and uses is more or less comparable to that already achieved upon using the synergistic mixture of document (5).

1.3.2 The Appellant has argued, instead, that the long term stabilization described in the patent in suit would be superior to the four weeks achieved in example 19 of document (5). In the Appellant's opinion this could be derived from the figures well above 100 in the column with headings "Time d" of Table 5 of the patent in suit, since these figures would indicate the number of days for which the stability of the slurries was tested. The stability achieved over several months by the claimed PAA/GWS methods and uses was also confirmed by the test report in document (7).

The Board notes however that even in the hypothetical case that the Appellant's interpretation of Table 5 of the patent in suit were plausible and the data of document (7) consistent, still it remains a fact that the patent itself expressly acknowledges e.g. in paragraph [0030] a testing time of not more than 11 days sufficient for determining "a clear long term effect".

Hence, the patent itself confirms that the "long term" stability achieved by the claimed PAA/GWS methods and uses can be substantially the same as the "prolonged" biocidal effect mentioned e.g. at column 2, lines 11 to 12, of document (5) and exemplified by the four weeks stability achieved in example 19 of this citation.

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1.3.3 On the other hand, the Respondent has argued that some of the experimental data described in the patent in suit and of those reported in document (7) would jeopardize the credibility of the achievement of antimicrobial inhibition over the whole scope of the claimed methods and uses.

The Board notes however that, taking into account the complexity of slurry contamination and microbial proliferation, some experimental discrepancies appear not necessarily conclusive for the presence within the claimed subject-matter of a substantial number of embodiments thereof providing unacceptably extensive microbial growth e.g. already within the first 11 days.

- 1.3.4 Hence, the Board finds, on the one side, that the claimed uses and methods and, thus, also the claimed PAA/GWS methods and uses actually achieve the aimed advantageous technical effect "a)" identified above at point 1.1, and, on the other side, that such effect is substantially the same stability over few weeks already achieved by using the synergistic mixture of example 19 in document (5).
- 1.3.5 In respect of the reduction of environmental concerns associated to the claimed methods and uses, i.e. the technical problem "b)", the Board notes that it is undisputed among the parties that PAA appears to the skilled person as an ingredient manifestly raising less concerns for the environment than the compounds conventionally used as non-oxidizing biocides. Hence the Board has no reason to doubt that the use of PAA as the sole biocide at the relatively large amounts required

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for the claimed PAA/GWS methods and uses, still produces less harm to the environment than the synergistic mixture of document (5) wherein non-oxidizing biocides are additionally present (although only in very small amounts).

1.3.6 Therefore, the Board concludes that the claimed PAA/GWS methods and uses provide vis-à-vis the prior art only the additional advantageous technical effect "b)" identified above at point 1.1.

Accordingly the technical problem credibly solved by the claimed PAA/GWS methods and uses vis-à-vis the method for prolonged microbial growth inhibition in pigment slurries already disclosed in example 19 of document (5), is that of rising less concerns for the environment.

1.4 In the opinion of the Board, it appears manifestly obvious for the skilled person to solve the posed problem by using in such example 19, instead of the synergistic mixture comprising the non-oxidizing biocide, any other compound that is already known to be effective as biocide in pigment slurries and known to rise less environmental concerns than the conventional non-oxidizing biocides.

The Board notes that already document (5) indisputably reminds the skilled reader that PAA alone has been used as biocide, e.g. in the food industry, because of its ability to enter the bacterial cell (see column 4, lines 41 to 65). In addition, the whole disclosure of document (5) - inclusive of the reference tests in all the examples based on PAA as the sole biocide - teaches that PAA alone acts, for at least a certain time at the

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concentrations used, as a biocide also in pigment containing slurries that are freshly prepared for or recovered during the papermaking process.

Additionally, as also conceded by the Appellant, to increase the initial concentration of a biocide represents an obvious measure for prolonging the time for which the antimicrobial effects of such ingredient remain detectable.

Finally, as discussed already above, it is undisputed among the parties that the skilled person would be aware that PAA rises less concerns in respect of the environment than the conventional biocides.

Hence, the skilled reader of document (5) that aims at avoiding the conventional non-oxidizing biocides considered harmful for the environment used in the synergistic mixture disclosed in this citation, would expect that an additional amount of PAA could be used instead of such undesirable compounds so as to compensate the predictable loss of biocidal effect unavoidably associated to the removal of these latter. Accordingly, the skilled person would simply carry out some optimization experiments and determine the amount of PAA alone providing the expected biocidal effect for e.g. about 11 days or more in the pigment slurry of example 19.

Nor would any inventive ingenuity be necessary to the skilled reader of document (5) for predicting that the same combination of long term inhibition of microbial growth with less harm to the environment would also occur in case the pigment slurry, instead of being freshly prepared as in example 19 of this citation, is

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derived from waste waters. Indeed, similarly to the patent in suit, this citation appears to implicitly consider as equivalent the microbial inhibition required for "whitewaters" in examples 1 to 18 and that needed for freshly prepared pigment slurries such as that of example 19.

Finally, it is undisputed that (as also evident from document (10), see the one but last paragraph on the left column at page 612 and the last full sentence, right column of page 631) gypsum is normally present in e.g. the "whitewaters" formed during papermaking, and even the patent in suit does not attribute to PAA any special biocidal effect onto specifically gypsum. The statement in paragraph [0013], referred to by the Appellant, indicates that peracids can form other "more stable calcium, potassium, sodium, ammonium and other persalts from which active oxygen is released more slowly", i.e. proposes an hypothetical reaction path that applies in general to any pigment slurry, and not only to the case of gypsum that is mentioned therein only as an example (see the last line of paragraph [0013] starting with "For example...").

- 1.4.1 The Appellant has nevertheless argued that the very same document (5) would lead away its skilled reader from the idea of using PAA as the sole biocide, since
 - the statement at column 2, lines 11 to 12, reading "To date, none of the commercially available biocides have exhibited a prolonged biocidal effect..."

as well as

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the reference tests in example 19

would indicate that PAA alone would not provide the biocidal effect for the required long term.

The Board notes firstly that the expression "the commercially available biocides" is not used in document (5) as inclusive of PAA as well (compare with e.g. column 4, lines 63 to 65). On the contrary, the statement at lines 11 to 12 of column 2 of this citation, when read in the context of the disclosure contained in the same column at lines 18 to 28, appears to only refer to the highly toxic and expensive commercially available biocides that were already conventionally used e.g. for waste water slurries of the paper industry.

Additionally, the reference tests reported in document (5) and based on PAA as the sole biocide are only indicative for the limited biocidal activity of this compound at the (low) concentrations used in such tests. Accordingly, also the experimental data reported for the reference tests in example 19 only suggest to the skilled person that PAA alone does not provide sufficiently long biocidal activity at the concentrations used in such tests.

In conclusion, this document teaches to the skilled person neither, in general, that it would be impossible to achieve sufficient biocidal effect lasting for few weeks when using PAA alone nor, in particular, that such biocide would be insufficient for long term stabilization even when used at concentrations

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appreciably higher than those used in the reference tests of example 19.

On the contrary, document (5) itself reminds the skilled reader at column 4, lines 53 to 57, that PAA is in general to be applied in high concentrations because it is an equilibrium molecule.

Hence, the skilled reader of example 19 of document (5) would still reasonably expect that the biocidal activity of PAA when used alone would last longer when used at higher concentration, e.g. at concentrations much higher of those used in this example.

- 1.4.2 The Board concludes, therefore, that the skilled person searching for a solution for the posed technical problem would have expected that PAA alone at sufficiently high concentration could be used instead of the synergistic mixture of example 19 of document (5) for providing long term biocide effect also to GWS, while avoiding the environmental concerns normally associated to the use of conventional non-oxidizing biocides. Hence, the skilled reader of this citation would have considered obvious to modify the prior art so as to arrive at the claimed PAA/GWS methods and uses.
- 1.5 Since, each claim 1 of any of the requests of the Appellant comprises either these obvious PAA/GWS methods or these obvious PAA/GWS uses, none of the Appellant's requests complies with Article 56 EPC 1973.

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Further issues

2. During the oral proceedings the Appellant has stated to be prepared to modify all its pending requests by introducing in each claim 1 a limit for the solid content of the slurry of at least 10% by weight. It has not argued that such restriction could be critical in order to ensure a specific additional technical advantage to the claimed subject-matter, but only that the introduction of this lower limit for the amount of solid would deprive of any possible relevance whatever technical teaching possibly derivable from example 16 of document (5), as this latter allegedly contained a very small amount of solids in the reference test wherein the microbial growth inhibition provided by PAA alone appeared superior to that obtained in the corresponding test containing the synergistic mixture.

The Board would like to stress in this respect that, as evident from the above reasoning, the Board's negative conclusions in respect of the Appellant's requests are not based specifically on the superior results for PAA as the sole biocide reported in example 16 of such citation. Therefore, the above reasons rendering obvious the presently claimed subject-matter would identically apply even in the case in which the claimed PAA/GWS methods and uses would had been further limited by requiring the gypsum to be present in the slurry in an amount of at least 10% by weight.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

G. Rauh

P.-P. Bracke