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D E C I S I O N
of 9 March 2004

Case Number: T 0695/01 - 3.3.5

Application Number: 93500111.5

Publication Number: 0635464

IPC: C04B 33/04

Language of the proceedings: EN

Title of invention:
A raw material composition for ceramic materials and process for its preparation

Patentee:
KAO CORPORATION, S.A.

Opponents:
ASSOPIASTRELLE
CERAMCO S.P.A.
LAMBERTI SpA
ASCER - Asociación Espanola Fabricantes de Azulejos, Pavimentos y Baldosas Cerámicas

Headword:
Ceramic composition/KAO

Relevant legal provisions:
EPC Art. 56

Keyword:
"Inventive step - no, obvious alternative"

Decisions cited:
-

Catchword:
-

Case Number: T 0695/01 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 9 March 2004

Appellant: (Proprietor of the patent)	KAO CORPORATION, S.A. Puig del Tudons, 10 ES-08210 Barbera del Valles (Barcelona) (ES)
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Representative:	Ungria Lopez, Javier c/o UNGRIA Patentes y Marcas, S.A. Avda. Ramon y Cajal, 78 E-28043 Madrid (ES)
Decision under appeal:	**Decision of the Opposition Division of the European Patent Office posted 18 April 2001 revoking European patent No. 0635464 pursuant to Article 102(1) EPC.**

Composition of the Board:

Chairman: M. M. Eberhard
Members: G. J. Wassenaar
H. Preglau

Summary of Facts and Submissions

I. European patent No. 0 635 464, was granted with 11 claims. Notices of opposition were filed by the four respondents (opponents). The opposition grounds were lack of novelty, lack of inventive step, unallowable amendments and insufficient disclosure. The claims as granted were amended. Three sets of amended claims were submitted during oral proceedings on 22 February 2001. Amended independent claim 1 according to the main request of the contested decision read as follows:

"An aqueous slurry composition for ceramics which comprises:
a.- From 50% to 80% of mineral clay having the following composition:
SiO_2 30-75%
Al_2O_3 13-35%
Fe_2O_3 4-8%
CaO 0.5-25%
MgO 0.2-3%
Na_2O 0.1-0.5%
K_2O 3-7%
TiO_2 0.2-1.5%
b.- From 0.02% to 2.75% by weight of water soluble acrylic polymer selected from the group consisting of (i) - (iii):
i.- Potassium, sodium, or ammonium polyacrylate having molecular weight from 2000 to 30000;
ii.- Potassium, sodium, or ammonium salt of copolymer of acrylic acid and methacrylic acid having molecular weight from 8000 to 30000;

iii.- Potassium, sodium, or ammonium salt of copolymer of acrylic acid and maleic acid having molecular weight from 5000 to 25000.
c.- From 0.01% to 2.25% by weight of inorganic alkali compound.
d.- From 20% to 50% of water; and
which has a pH value of 9.2 to 10.3 and a viscosity of $\leqslant$ 1800 cps."

Claim 1 of the first auxiliary request differed from claim 1 of the main request only in that the viscosity range was narrowed to less than 1000 cps.

II. The opposition division held that the amendments in claim 1 of the main request fulfilled the requirements of Article 123(2) and (3) EPC and that the invention was sufficiently disclosed within the meaning of Article 83 EPC. The patent was revoked on the ground that the subject-matter of claim 1 of all the requests lacked an inventive step within the meaning of Article 56 EPC. The reasons were supported, inter alia, by the following documents:

D2: Ceramica Informazione, Feb. 1976, No. 114, pages 71-74, "Il ruolo degli agenti disperdenti nella fluidificazione degli impasti ceramici", by B. Soldavini. (+ English translation, pages 1-8,)

D5: US-A-4 742 105
D6: US-A-4 186 027
D7: Ceramica Informazione, 1991, No, 302 and 303, pages 273-282 and 337-344, "Evoluzione e sviluppo della tecnologia di fabbricazione della piastrelle per interni dalla bicottura alla monocottura rapida", by L.

Contoli and A. Brusa (+ English translation, pages 16-30).

D11: Documentation of Fratelli Lambert SpA concerning "REOTAN LPS-LP/4".

III. The appellant (proprietor) lodged an appeal against this decision. With the statement of the grounds of appeal dated 23 August 2001, the appellant filed a new set of claims as its main request and submitted new comparative examples to demonstrate the improved mechanical strength of the green bodies resulting from a slurry composition according to claim 1 on file. This claim was identical to claim 1 of the main request on which the contested decision was based. The appellant further submitted the following two documents:

P3: Grant T. Wernimont, "Use of statistics to develop and evaluate analytical methods", Association of Official Analytical Chemists, pages 28-31 and 150-153.

P4: José Luis Amoros et al, "Manual para el control de la calidad de materias primas arcillosas", Instituto de Technologia Cerámica, 1998, pages 51-57.

During oral proceedings, which took place on 9 March 2004, a further document and a new set of claims as auxiliary request were submitted. Claim 1 of the auxiliary request differed from claim 1 of the main request only in that the viscosity range was limited to "$\leqslant$ 1000 cps".
The new document was a page from an undated publication by SACMI called "Rivestimento/Wall tiles", hereinafter referred to as P5.

IV. The arguments of the appellant with respect to inventive step can be summarized as follows:

Starting from D2 as the closest prior art document, the problem to be solved was to provide an aqueous clay slurry for ceramics of which tiles could be produced having improved mechanical strength. It was surprisingly found that the type of clay and the selection of the pH range as claimed solved this problem. The effect was proved by comparative examples filed during the examination proceedings with the letter dated 11 September 1997 and the comparative examples filed with the grounds of appeal. None of the prior art documents suggested the claimed choice of clay and pH range, let alone their positive effect on the mechanical strength of the ceramic tiles produced thereof. With respect to tile production D2 indicated to use traditional deflocculants rather than synthetic organic fluidifying agents. It could not be derived from D7 or any other citation that the use of red clays with high iron content would improve the mechanical strength. Also P5 confirmed that it was not known that the use of red clays would improve the mechanical strength.

V. The respondents (opponents) refuted the arguments of the appellant and, apart from the novelty objection, maintained their objections raised in the proceedings before the first instance. The arguments of the respondents with respect to inventive step may be summarized as follows.

Neither the patent in suit nor the comparative examples supported the appellant's claim that the choice of clay and pH range improved the mechanical strength of the tiles. The comparative examples were not properly executed and the statistical evaluation was not correct. Moreover, the critical parameter for the mechanical strength was the particle size distribution in the slurry. This essential feature was however not disclosed. It followed from D11 that the deflocculant "REOTAN LP/4", used in D2 in an amount of 0.1 to 0.3 %, was an aqueous mixture of polyacrylic acid and sodium hydroxide in agreement with present claim 1. Similar aqueous mixtures were also disclosed in D5 and D6. In view of D7, disclosing that iron containing clay for red stoneware had the highest green strength, it was obvious to use red clay. Moreover red clay was the traditional clay for making ceramic tiles. White clays were only recently used for that purpose. In view of Figure 5 of D2 and the teaching of D6 and/or D5 it was obvious to choose a pH in the claimed range.

VI. The appellant requested that the decision under appeal be set aside and the patent be maintained on the basis of the set of claims filed with letter dated 23 August 2001 (main request) or on the basis of the set of claims filed during the oral proceedings (auxiliary request).

The respondents requested that the appeal be dismissed.

Reasons for the Decision

1. Claim 1 of the present main request is identical to claim 1 of the main request on which the contested

decision is based. The opposition division held that the amendments in claim 1 fulfilled the requirements of Article 123(2) and (3) EPC. They also held that the invention was sufficiently disclosed within the meaning of Article 83 EPC. The respondents maintained their objections under Article 100(b) and (c) EPC but their arguments could not convince the board that the findings of the opposition division in this respect should be reversed. Because the final decision reached in this case allows the request to dismiss the appeal by the respondents, it is not necessary to provide further reasons for the board's conclusion in these matters.

2. The slurry composition according to claim 1 of the main request is new. Novelty is in fact no longer in dispute. In agreement with the appellant, the board considers that D2 is the closest prior art document with respect to the subject-matter of claim 1. D2 relates to the role of dispersing or deflocculating agents in aqueous ceramic slurries. It is observed therein that the polymeric organic dispersants are superior in their property to maintain a low viscosity of the slurry at high dry content for a longer period compared with the more traditional inorganic fluidifying agents such as sodium silicate, carbonate and polyphosphates (page 1 of the English translation). A dry content of between 60% and 80% by weight and a concentration of for example 0.1% of deflocculant to the dry material are disclosed (page 3, 2nd paragraph of the English translation). With respect to tile production D2 does indeed also indicate that, concerning only the fluidifying aspect of the products, at present the traditional deflocculants, sodium carbonate, silicate

and polyphosphates, may be regarded as the most convenient, and that in fact their lower unit cost compensates for the need to use higher doses than required for organic fluidifying agents (English translation, page 7, last line to page 8, 1st and 2nd paragraphs). This passage is, however, followed by the observation that of course this is a general rule, because there are many examples in which the required fluidifying results can only be obtained using the latter products. Since D2 also comprises an example in which a synthetic organic deflocculant is used in a slurry to be atomised for the production of tiles, as illustrated in Figure 3, there can be no doubt that D2 clearly and unambiguously discloses the use of synthetic organic deflocculants as an alternative for traditional deflocculants in the production of tiles.

3. D2 further discloses that the dispersing agents are all electrolytes or polyelectrolytes in the form of sodium or ammonium salts, easily soluble in water and that polyacrylic organic deflocculating agents are sold under the trade name REOTAN (page 2, 3rd and 8th paragraphs and page 6, 1st paragraph of the English translation). D2 also discloses that the concentration of the deflocculant and the pH of the slurry have considerable influence upon the viscosity of the slurry (page 4, 3rd paragraph of the English translation). Figure 3 of D2 shows the influence of the concentration of "REOTAN LP/4" upon the viscosity of a slip of white paste with a specific weight of 1786 g/l intended for atomising to produce tiles. Below about 0.15% the viscosity is above 3000 cps, whereas at around 0.2% the viscosity is below 500 cps (page 4, 2nd paragraph, of the English translation and Figure 3).

4. According to the submissions of the appellant during oral proceedings, starting from D2, the problem to be solved would have been to provide an aqueous slurry for ceramics from which ceramic tiles having improved mechanical strength may be produced. The inventors would have surprisingly found that the combination of the type of clay and the pH range as claimed improved the mechanical strength of the tiles. The board can, however, not accept these submissions as a basis for defining the objective problem underlying the invention for the following reasons.

5. According to the patent in suit it has indeed originally been an object of the invention to improve the mechanical strength of the green and fired tiles, but this improvement has been solely attributed to the presence of the mixture of polymer and alkali (components b and c of claim 1) instead of conventional dispersing agents; see page 3, lines 20 to 26 and Example 3. With respect to the selected clay composition the patent discloses only that it is a preferred composition, without any reference to its effect (page 3, lines 32 to 43). With respect to the selected pH range it is indicated that it maximizes the degree of dispersion by avoiding the pH zero point of charge of the clay components (page 4, line 58 to page 5, line 6). Example 3 of the patent in suit, which reports an increase in the green mechanical strength (see page 9, Table and lines 54 to 55), was performed using on the one hand the claimed mixture of polymer and alkali (batch 2) and on the other hand a conventional dispersing agent (sodium tripolyphosphate + sodium metasilicate: batch 1). Therefore, this

example does not illustrate an improvement of the mechanical strength of the green and/or fired tiles with respect to the closest prior art D2, which teaches the use of polyacrylic organic deflocculating agents.

6. The effect of the clay composition on the mechanical properties of the tiles was submitted for the first time in the letter dated 11 September 1997, filed during the examination proceedings. This letter comprises a comparative example of a clay according to present claim 1 (Kao clay) and a ball clay composition, which largely corresponds to ball clay C published in "Ceramic Industry", January 1996 on page 64. The green body mechanical strength of the tile produced with the Kao clay is indicated to be higher (21.9 kg/cm^2) than that of the ball clay (17.5 kg/cm^2). The board does not dispute these results and considers that it is credible that the clay composition has an effect on the mechanical properties of the products made thereof. The comparative examples, however, do not prove that clay compositions according to claim 1 generally result in ceramic tiles having improved mechanical strength compared with ceramic composition normally used for the production of ceramic tiles by atomizing the slurry and pressing the powder composition. It was submitted by the respondents during oral proceedings that ball clays were not, or only to a limited amount, used for that purpose. In the article concerning ball clays on the cited page in Ceramic Industry it is indicated that the amount of ball clay commonly used in floor tiles is 0-25% and in wall tiles 25-40% (end of said article). Thus the comparison with a composition containing as the clay component essentially a ball clay is not suitable to demonstrate improved mechanical strength

with respect to clay compositions normally used for ceramic tiles. Moreover, the comparison has only been made with one specific Kao clay. The composition ranges for the main components of the clay in claim 1 are, however, extremely broad so that it comprises very different mineralogical compositions which may have very different chemical properties and may therefore interact differently with the deflocculant, providing products with different mechanical properties. In particular the calcium oxide content of the clay according to claim 1 may vary largely between 0.5 and 25%. It follows from D7 that the calcium oxide content in the form of calcite has a large influence on the mechanical strength; see English translation, page 16, right hand column and tables 3 to 5 and 10 to 12. It has thus not been made credible that an improvement in mechanical strength could be obtained over the whole range of the claim.

7. The effect of the pH range on the mechanical strength has been submitted for the first time in the grounds of appeal, comprising further comparative examples. According to these examples the green mechanical strength at pH 9.7 has an average value of 32.0 kg/cm^2, whereas at pH 8.5 and 11.0 the average value is 29.3 kg/cm^2. The experiments and the results have been heavily criticized by the respondents and the board has serious doubts about the relevance of these new comparative examples. Even if it were assumed in favour of the appellant that the choice of the pH within the claimed range actually results in an improvement in mechanical strength for the specific clay composition tested, this improvement is small. However there is no evidence that an improvement would also be observed for

other clay compositions falling under claim 1. Considering the small increase obtained with the tested clay and the relatively low accuracy of the measurement method, on the one hand, and the fact that claim 1 encompasses clay compositions which may have very different mineralogical compositions, on the other hand, the board is not convinced, in the absence of tests with other clay compositions, that an improvement of the mechanical strength by choosing a pH within the claimed range is actually achieved over the whole range of claim 1. See also the reasons given in the 2nd part of point 6, which apply likewise.

8. It follows from the above that starting from D2 the problem underlying the invention can only be seen in providing further aqueous slurry compositions suitable for the manufacture of ceramic tiles. The appellant proposes to solve this problem by a slurry composition according to claim 1. On the basis of Example 3 of the patent in suit it is credible that such a composition actually solves that problem. The composition according to claim 1 differs from those disclosed in D2 in the composition of the clay, the selection of the acrylic polymer, the addition of an inorganic alkali compound and the selection of the pH range.

9. The only ceramic composition for the production of tiles mentioned in D2 is a white paste of unknown composition. Since it is a white paste it cannot comprise a mineral clay according to claim 1, which, because of its high iron content, is a red clay. The use of red clays is, however, common in the art of tile production, especially for floor tiles. See in this respect D7, paragraphs 2.1.2 and 2.2, wherein red and

white clay compositions for tiles are discussed. For red stoneware two clay compositions have been disclosed in table 10, which differ from the composition according to claim 1 only in a slightly higher sodium content (see samples 1 and 2). Sample 2 has a sodium oxide content of 1.18%, whereas claim 1 requires 0.1 to 0.5% Na_2O. For clays, being natural products, such slight variations cannot be considered as significant in the absence of evidence to the contrary. In the present case no effect due to the rather low amount of sodium has been made credible. The appellant alleged at the oral proceedings that the presence of the specific amount of Na_2O (0.1 - 0.5%) in combination with the claimed amount of Fe_2O_3 (4-8%) had an influence on the mechanical strength of the green bodies. This was however strongly disputed by the respondents. In the absence of any evidence from the appellant, who has the burden of proof for this alleged effect, the appellant's allegation cannot be accepted. In view of the statements in the patent in suit with respect to the clay compositions it appears that the limitations of the clay composition in claim 1 are just a consequence of the compositions of the local Spanish clays available to the appellant (page 3, lines 32 to 43). In fact, in the invention as originally presented, no particular attention was paid to the clay composition. None of the ten originally filed claims comprised any limitation to the mineral clay to be used in the slurry composition. The slight deviation in chemical composition of the clay according to claim 1, which has not been shown to have a technical effect, does not involve an inventive step.

10. The polyacrylic deflocculant used in D2 is REOTAN LP/4. Respondent 3 provided evidence (D11) concerning the composition of this deflocculant. In the board's view, however, D11 does not unambiguously prove that REOTAN LP/4 used in D2, actually comprised a sodium polyacrylate according to feature b(i) of claim 1 and an inorganic alkali compound according to feature c of claim 1. Low molecular weight sodium polyacrylates are, however, known as dispersants for clay slurries; see in this respect D5 (column 1, line 53 to column 2, line 24 and claims 1, 4 and 6). The board does not dispute that D5 relates to koalinitic clay slurries for paper coating. It is, however, undisputed that it was common general knowledge before the filing date of the patent in suit that clays were not only used for ceramics but also for various other purposes such as the coating of paper and their use as filling materials in plastics and rubber products. The skilled person in the art of tile production was also aware that the problem of providing high solid clay slurries with a low viscosity is not limited to clay slurries for the production of tiles and that the slurry rheology is common to all concentrated clay suspensions. For the atomizing step in the tile production good stability and pumpability is required. The skilled person would thus have also considered documents relating to clay slurries where the same requirements are important such as clay suspensions used for paper coating. D5 relates to such problems (column 1, lines 35 to 49). It discloses binary mixtures of polyacrylates and sodium silicates as deflocculating agents for kaolin slurries. These mixtures contain at least one of sodium, potassium and ammonium polyacrylate having a weight average molecular weight of between 2000 and about 10 000, i.e. a

molecular weight falling within the claimed range, and sodium silicate. These components are present in amounts of greater than 50 to 80 wt.% and less than 50 to 20 wt.% on a dry solid base respectively. The polyacrylate is essentially neutralised, sodium polyacrylate being generally used. The sodium silicates are for example commercial sodium silicates such as $Na_2O:2SiO_2$, sodium orthosilicate and sodium metasilicate (see claims 1 and 6; column 2, lines 17 to 32). The deflocculating mixture is used in an amount of 0.4 to 1% by weight on a dry solid basis in the slurry (claim 7). D5 discloses as an example a mixture of 67% by weight sodium polyacrylate having a weight average molecular weight of 4200 and 33% by weight sodium silicate ($Na_2O:2SiO_2$) as deflocculating agent (abstract, claims 2 and 9 and column 2, lines 38 to 42). Table II discloses a kaolin slurry having a solid weight of 58% comprising an amount of 0.50% of such a mixture, calculated on a solid basis. Thus, apart from the chemical composition of the clay, such a slurry fulfils all the requirements of features a, b and c of present claim 1. D5 further teaches that the use of the binary mixture to reduce the viscosity of kaolin slurries is in most instances superior to the use of each component alone (column 4, lines 26 to 29). The viscosity values reported in D5 are well below the upper limit of 1800 cps stated in present claim 1. In the board's judgment a skilled person would in view of this teaching have considered the nature and the amount of the deflocculant used in D5 also for preparing stable and pumpable slurries to be atomized for the production of tiles.

11. It is already known from D2 that the effect of the deflocculant on the viscosity of the slurry is dependent upon the pH of the slurry (page 4, 3rd paragraph of the English translation). D2 specifically discloses pH values of 7.8 and 9.3 (Figure 5). D5 does not disclose the pH of the slurry but considering the presence of sodium silicate, and the fact that the polyacrylate is neutralised, it is very likely that most of the slurries have a pH value above 7. The optimum pH value is of course dependent upon the amount and the nature of the clay and the deflocculant. It is also immediately apparent to the skilled person that the pH of the slurry will be changed by varying the amount of the binary deflocculant in the clay slurry and/or the relative amount of the sodium silicate in the binary deflocculant known from D5. In view of the teaching of D2 and D5, the determination of the optimum pH value leading to the appropriate viscosity for atomizing the slurry is a matter of routine experimentation. In the absence of any surprising effect no inventive step can therefore be seen in the choice of the pH range of 9.2 to 10.3 according to claim 1.

12. For these reasons the board holds that the subject-matter of claim 1 of the main request does not involve an inventive step within the meaning of Article 56 EPC. Since D2 already discloses optimum viscosity values well below 1000 cps the same applies to the subject-matter of claim 1 of the auxiliary request. The documents P3 to P5, relied on by the appellant in the appeal proceedings do not contain any additional information which could change the preceding considerations concerning the lack of inventive step.

The above reasons for lack of inventive step being mainly supported by three documents (D2, D5 and D7), the appellant's argument in writing that the solution of the technical problem is not obvious in a case where the skilled person has to combine the teachings of four or more different references, fails.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar: The Chairman:

G. Rauh M. M. Eberhard