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D E C I S I O N
of 1 February 2000

Case Number: T 0548/95 - 3.3.5

Application Number: 84308176.1

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Language of the proceedings: EN

Title of invention:

Non-expansive, rapid setting cement

Patentee:

UNITED STATES GYPSUM COMPANY

Opponent:

Ardex-Chemie GmbH

Headword:

Cement/USGC

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step (no)"

Decisions cited:

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Catchword:

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Boards of Appeal

Chambres de recours

Case Number: T 0548/95 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 1 February 2000

Appellant: UNITED STATES GYPSUM COMPANY
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 25 April 1995
revoking European patent No. 0 143 633 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: R. K. Spangenberg
Members: M. M. Eberhard
M. B. Günzel

Summary of Facts and Submissions

- I. European patent No. 0 143 633 based on application No. 84 308 176.1 was granted on the basis of 13 claims. Granted claims 1 and 7 read as follows:

"1. A non-expansive, ettringite-producing hydraulic cement which produces a cementitious product which is resistant to carbon dioxide attack, is stable at high temperatures and has a high early strength, said hydraulic cement comprising from 72% to 80% portland cement, from 14% to 21% high alumina cement having a surface area of 3000 to 9000 cm²/g, from 3.5% to 10% calcium sulfate and from 0.4% to 0.7% hydrated lime, by weight."

"7. A non-expansive, ettringite-containing cementitious composition comprising the hydration products of a hydraulic cement comprising from 72% to 80% portland cement, from 14% to 21% high alumina cement having a surface area of 3000 to 9000 cm²/g, from 3.5% to 10% calcium sulfate and from 0.4% to 0.7% hydrated lime, by weight."

- II. The respondent (opponent) filed a notice of opposition requesting revocation of the patent on the ground of lack of inventive step. Later on, he set forth two further grounds of opposition, ie lack of novelty and insufficiency of disclosure. The respondent relied inter alia on US-A-3 997 353 (D3) and US-A-4 350 533 (D4) in support of his arguments. The appellant (patentee) filed an amended set of claims, as an auxiliary request, with a letter dated 28 June 1994. The claims of this request differ from the granted

claims only in that claims 7 to 13 were deleted.

III. The opposition division revoked the patent on the grounds of lack of novelty and lack of inventive step. It took the view that the cementitious composition according to claim 7 as granted lacked novelty over the hydrated product resulting from the cement mixture disclosed in example IIA of D3. It further held that the hydraulic cement according to claim 1 of the auxiliary request did not involve an inventive step in view of the teaching of D3 and D4 and pointed out inter alia that no evidence had been provided that the addition of hydrated lime as an extraneous component was critical.

IV. The appellant lodged an appeal against this decision. The appellant's arguments in the statement of grounds of appeal can be summarised as follows:

The cement according to claim 1 had a high early strength and was thus especially useful in the high speed production of cement board products. Moreover, it provided hydrated products which were stable at high temperatures and resistant to carbon dioxide attack due to their reduced amount of ettringite. The cement disclosed in D3 had less Portland cement and more calcium aluminate than the claimed cement. Sample A in example II of D3 contained Portland cement, calcium aluminate and calcium sulphate all within the claimed ranges of the patent in suit; however, the cement mixture did not fall within the scope of claim 1 because it lacked added hydrated lime. Sample A developed very low early strength of about 80 psi after two hours. In contrast thereto, the compressive

strength of the claimed compositions was in excess of 2000 psi within one hour. Thus, example II A of D3 was a clear teaching away from the claimed cement. This lack of high early strength arose because sample A contained no hydrated lime added to the other ingredients. There was no teaching in D3 relating to resistance to carbon dioxide attack and stability of the cementitious product at high temperatures. D3 disclosed that the presence of free lime in an amount greater than 2 wt.% of the Portland cement had a deleterious effect. It did not teach incorporating hydrated lime into a hydraulic cement comprising portland cement. In fact it taught the opposite, namely that there should be a substantial absence of free lime in order to provide the desired results. An absence of free lime was defined as less than 0.2 wt%. In contrast at least 0.4 wt% were required in claim 1. D3 would have led the skilled person away from having any free lime present in the cement and, in view of the compressive strengths in D3, the high early strength attained with the claimed cements was clearly unexpected.

D4 disclosed cement compositions which required much higher amounts of calcium sulphate and high-alumina cement than the claimed cement. Thus, much higher amounts of ettringite were formed on hydration. The high ettringite content made the hydrated products unstable at high temperatures and susceptible to degradation by carbon dioxide. The purpose of the present invention was the formation of much lower amounts of ettringite in order to achieve the desired resistance to carbon dioxide attack and stability at high temperatures. D4 was concerned with the total

amount of ettringite formed during the hydration reaction and not with the rate of formation of ettringite which was critical to the claimed invention. Since the claimed composition required the rapid formation of ettringite to provide early strength to manufacture cement boards, the added lime had to be in its most reactive form and was incorporated into the composition as hydrated lime. The free calcium oxide present in the Portland cement was not sufficiently reactive to initiate the rapid formation of ettringite and to provide the early strength required for high speed production of cement boards. Extraneous lime was not required in D4 and where it was used, the amounts were much greater than the amounts of added hydrated lime stated in claim 1. Moreover, D4 was concerned with an expansive cement, whereas claim 1 related to a non-expansive cement.

V. The respondent put forward inter alia the following arguments:

The appellant's submissions about the effect of hydrated lime on the formation of ettringite were not correct. D4 taught on the contrary that the free lime contained in the Portland cement and extraneous lime added in the form of quick lime or hydrated lime had the same influence on the formation of ettringite. Furthermore, the respondent's comparative tests filed with the letter of 21 April 1994 (hereinafter D10) showed that the addition of hydrated lime in the amounts stated in claim 1 to a composition containing Portland cement, aluminous cement and calcium sulphate had no influence on the development of compressive and flexural strength. They also demonstrated that the said

addition had no influence on the formation of ettringite after a curing time of 5 and 24 hours and that the early strength depended considerably on the kind of Portland cement. They further proved that the high values of early strength in the examples of the patent in suit were not caused by the addition of hydrated lime in the amounts stated in claim 1 but resulted from the tests being performed under other operating conditions and from the use of specific additives. As the obtention of these high values depended in a considerable extent on other parameters which were not disclosed in the patent in suit, the disclosure of the patent in suit did not meet the requirement of sufficiency of disclosure.

The subject-matter of claim 1 did not involve an inventive step. The cement mixture of sample A in example II of D3 represented the closest prior art. The comparative tests D10 showed that the addition of 0.4 to 0.7 wt.% hydrated lime had no effect on the early strength and on the rate of ettringite formation. The appellant had not shown that the addition of hydrated lime enhanced the resistance to carbon dioxide and/or the temperature stability of the products produced with the claimed cement. Therefore, it could not be discerned what technical problem had been solved by this feature with respect to the closest prior art. In view of the teaching of D3 about the maximum content of free lime in the cement mixture, it was obvious for the skilled person to limit the amount of free lime. Furthermore, D4 taught that free lime contained in the Portland cement and hydrated lime were alternative materials and, thus, gave the skilled person an incentive to replace a part of the calcium oxide

required for the formation of ettringite by hydrated lime. It did not matter whether the small amount of lime was added to the cement mixture or was brought by the other ingredients thereof.

VI. In a communication from the board, doubts were expressed concerning the novelty of the composition according to claim 7 of the main request. Regarding claim 1 of both requests, the board informed the parties of its provisional opinion about inventive step, namely that the subject-matter of claim 1 appeared to lack an inventive step over the teaching of D3 and D4, and gave the reasons in support of this opinion. In this context, it was referred to the common general knowledge about the fineness of high-alumina cements as illustrated by "The Chemistry of Cement and Concrete", F. Lea, 1970, page 510 (hereinafter D11). The appellant did not reply to this communication.

VII. The appellant requested in the statement of grounds of appeal that the decision under appeal be set aside and that the patent be maintained as granted. As an auxiliary request, the appellant requested that the patent be maintained with claims 1 to 6 according to the auxiliary request submitted with the letter of 28 June 1994. The respondent requested that the patent be revoked, ie that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

Main request (granted claims)

2. The cement as defined in claim 1 of the main request is novel with respect to the cement mixture disclosed in example II A of D3 in that it comprises 0.4 to 0.7 wt% hydrated lime. It is assumed to the appellant's benefit that the hydrated lime is added as a separate ingredient. In the absence of evidence showing that the calcium aluminate used in the said example has a surface area falling inevitably within the range stated in claim 1, this feature is also considered to be new over the disclosure of D3. The additional features in claim 1 that the hydraulic cement produces a cementitious product which is stable at high temperatures and has a high early strength cannot be considered as distinguishing features in the absence of precise data defining the relative terms "high" and "early" in this claim. The same applies to the resistance to carbon dioxide attack since claim 1 contains no precise data defining this resistance. It was not disputed that the subject-matter of claim 1 is new over the disclosure of the other documents cited in the opposition proceedings.

The question arises whether or not the subject-matter of claim 7 of the main request is novel over the disclosure of D3 (example II, sample A). However, as claim 1 of this request is not allowable for the reasons given hereinafter, this issue need not be decided.

3. According to the respondent, D3 and more specifically the cement mixture disclosed in example II A thereof represents the closest prior art. The composition of

this cement mixture is closer to the claimed subject-matter than all the remaining compositions indicated in D3. This was not contested by the appellant. In these circumstances, the board can accept that example II A be taken as starting point for the definition of the technical problem underlying the claimed cement although the hydration product obtained in this example has a much lower early compressive strength than most of the other cement mixtures disclosed in D3.

3.1 D3 discloses a cement mixture containing 75 wt% Portland cement (type I), 19.5 wt% calcium aluminate and 5.5 wt% calcium sulphate hemi-hydrate (see example II, sample A). The calcium aluminate added to the Portland cement comprises CaO and Al₂O₃ in a stoichiometric ratio of one part CaO to 0.58-2.5 parts Al₂O₃. It is a burned or reacted compound having a free lime content of less than 0.2 wt%, for example a calcium aluminate clinker (see column 4, lines 30 to 33; column 5, lines 1 to 6; column 6, line 25). The calcium aluminate is in the form of ground particles having a median particle size of 10-20 µm measured by a Coulter counter and 80% of the particles are smaller than 44 µm (column 5, lines 9 to 13). Sodium citrate was added to sample A as a retardant, in an amount of 0.2 wt.%. This sample was mixed with water and sand in a ratio of 26 parts cement, 74 parts sand and 11 parts water (parts by weight). The resulting product has a compressive strength of 0.55 MPa, 2.16 MPa and 31.7 MPa after 2 hours, 24 hours and 28 days respectively.

3.2 Concerning the technical problem solved by the hydraulic cement of claim 1 with respect to this prior art, the appellant contended that the claimed hydraulic

cement exhibited a higher early strength than the cement mixture of example II A. According to the appellant, the lack of high early strength in Example IIA of D3 arose because sample A contained no hydrated lime added to the other ingredients of the composition. Calcium oxide present in the Portland cement was slower to react than hydrated lime, so the appellant, and the addition of 0.4 to 0.7 wt% of hydrated lime to the other ingredients of the composition would lead to a higher early strength. The board cannot accept that the alleged improvement of the early compressive strength would be achieved for **all the hydraulic cements** encompassed by claim 1 for the following reasons:

It is not indicated in the patent in suit that the addition of this small amount of hydrated lime results in a higher early compressive strength and no evidence was provided to prove such an effect. Furthermore, this effect was contested by the respondent who has submitted comparative tests in support of his arguments with his letter dated 21 April 1994 (D10). The comparative tests D10 show that the addition of 0.4 to 0.7 wt% of hydrated lime to a composition containing 75 wt% Portland cement (PZ35F or PZ55F), 19.5 wt% high-alumina cement and 5.5 wt% calcium sulphate hemihydrate does not produce any substantial change of the compressive strength at an early age, ie after 5 hours (see Table 2). As regards the rate of formation of ettringite, a clear dependence of the rate of ettringite formation on the amount (0.4 to 1 wt.%) of added hydrated lime could not be observed as pointed out in D10 (see page 2). This was not contested by the appellant. In view of this experimental report which

was not contested by the appellant, the board is not convinced that the addition of 0.4 to 0.7 wt% of hydrated lime to a cement mixture containing Portland cement, high-alumina cement and calcium sulphate in the relative amounts stated in claim 1 or in Example IIA of D3 would lead to a higher early compressive strength. The higher values of early compressive strength obtained in examples 2 to 6 of the patent in suit with respect to example IIA of D3 would appear, in view of these comparative tests, to result from parameters or features not mentioned in claim 1. A direct comparison of the values of early compressive strength indicated in example IIA of D3 with those given in the patent in suit is not relevant since the examples of the patent in suit do not differ from example IIA only by the presence of added hydrated lime but also by a lot of other parameters such as the temperature of the mix water and of the solid components, the kind of Portland cement, the amount of cement in the mortar, the kind of additives (super-plasticiser and retardant), the curing temperature. As the appellant has himself argued that the considerably higher early strength of the claimed compositions results from the addition of hydrated lime into the mixture, the burden of proof lies on his side, all the more so as the respondent's comparative tests prove the contrary, at least for the kind of Portland cement used in these tests and in the absence of additives. Claim 1, however, covers any kind of Portland cement and both the presence or the absence of additives.

Furthermore, it has not been shown that the addition of hydrated lime to the cement mixture of Example IIA or to a cement mixture comprising Portland cement, a high-alumina cement and calcium sulphate in the relative amounts stated in the said example would improve the resistance of the hardened products to carbon dioxide degradation or their stability at high temperatures, or the setting time of the cement mixture.

In the absence of evidence showing an improvement over the non-expansive hydraulic cement of example IIA of D3, the technical problem underlying the claimed hydraulic cement can only be seen in the provision of an alternative non-expansive hydraulic cement. It is proposed that this problem be solved by the hydraulic cement as defined in claim 1, which differs from the cement mixture of example IIA of D3 in that it contains from 0.4 to 0.7 wt.% hydrated lime (as extraneous lime) and the high-alumina cement has a surface area of 3000 to 9000 cm²/g. It is credible and undisputed that the technical problem stated above has actually been solved by the claimed process.

- 3.3 Although D3 does not disclose the surface area of the calcium aluminate, it gives information about its average particle size as determined by a Coulter counter and about the advantages resulting from such a particle size. It further discloses that very fine particles cause flash setting of the cement while the use of coarser particles can result in undesirable expansion (see column 5, lines 5 to 24). Furthermore, high-alumina cements having a Blaine surface area of 2500 to 4000 cm²/g, commonly about 3000 cm²/g were well-known before the priority date (see D11, page 510,

paragraph titled "fineness"). In view of the teaching of D3 and taking into account the common general knowledge concerning high-alumina cements, it would have been obvious to the skilled person to choose a high-alumina cement having a surface area within the claimed range in order to solve the problem stated.

As regards the addition of extraneous hydrated lime, D3 does not teach adding extraneous hydrated lime into the cement mixture. However, it discloses that the Portland cement used in D3 contains not more than 2 wt% free calcium oxide and that the presence of free lime in an amount greater than 2 wt% of the portland cement in the cement mixture would have a deleterious effect (see column 3, lines 46 to 52; column 4, lines 4 to 16; column 8, table IV). Therefore, the skilled person was aware of the fact that the amount of free lime in the cement mixture had to be limited in order to avoid deterioration of the cement properties, as argued by the respondent. Concerning the appellant's arguments about the absence of free lime being defined as less than 0.2 wt.% CaO or Ca(OH)₂ in column 5, lines 4 to 8, the board observes that this value does not concern the amount of free lime present in the cement mixture but the free lime content of the calcium aluminate. The lime content of the cement mixture may be higher since up to 2 wt.% free lime may be present in the Portland cement (see column 4, lines 4 to 10).

Furthermore, D4 discloses high early strength, non-expansive cementitious compositions containing a high-alumina cement, calcium sulphate, Portland cement and optionally extraneous lime, which produce a certain amount of ettringite during the early stages of

hydration. According to D4, the lime necessary for ettringite formation in these compositions may come entirely from the Portland cement; alternatively some or all of the lime may be added as quick lime or slaked lime. The lime present in the Portland cement and extraneous lime (quick lime or slaked lime) are considered to be **alternative or complementary sources** of calcium oxide during hydration of the cement mixture (see column 3, lines 64 to 68; column 4, lines 1 to 2, 6 to 24 and 64 to 67). In view of this additional teaching, it would have been obvious to the skilled person faced with the problem stated above that the addition of small amounts of hydrated lime to a composition such as that of Example IIA of D3 would lead to an alternative non-expansive hydraulic cement, provided the total amount of free lime in the cement mixture is not greater than 2% by weight of the Portland cement in order to avoid the deleterious effect on the compressive strength disclosed in D3 (see column 4, lines 7 to 10 and column 8, Table IV). The appellant's argument that D4 was concerned with an expansive cement contrary to the claimed cement, is not convincing since one of the objectives of D4 is "to provide a non-expansive cementitious composition in which ettringite comprises from about 2% to about 60% of the total weight" (see column 3, line 67 to column 4, line 2) and D4 teaches how to avoid expansion in column 5, second paragraph.

- 3.4 It follows from the above that the hydraulic cement according to claim 1 of the main request does not involve an inventive step. As claim 1 does not meet the requirement of inventive step set out in Articles 52(1) and 56 EPC, it is not allowable and the main request

must fail.

Auxiliary request

4. Claim 1 of the auxiliary request is identical to claim 1 of the main request. Therefore, the reasons indicated above in connection with the inventive step issue apply likewise to claim 1 of the auxiliary request. Thus, the subject-matter of claim 1 of the auxiliary request lacks an inventive step and this request must also fail.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

S. Hue

R. Spangenberg