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D E C I S I O N
of 20 September 2000

Case Number: T 0970/97 - 3.3.5

Application Number: 90200664.2

Publication Number: 0389061

IPC: C01F 7/04

Language of the proceedings: EN

Title of invention:

Process for the production of aluminium hydroxide from bauxite

Patentee:

SHELL INTERNATIONALE RESEARCH MAATSHCAPPIJ B.V.

Opponent:

ALCAN INTERNATIONAL LIMITED

Headword:

Bauxite digestion/SHELL

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step - yes"
"Non-obvious solution of a technical problem"
"Time factor as secondary indication"

Decisions cited:

-

Catchword:

-



Case Number: T 0970/97 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 20 September 2000

Appellant: SHELL INTERNATIONALE RESEARCH
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Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 26 June 1997
revoking European patent No. 0 389 061 pursuant
to Article 102(1) EPC.

Composition of the Board:

Chairman: R. K. Spangenberg
Members: G. J. Wassenaar
J. H. Van Moer

Summary of Facts and Submissions

I. The appeal is from the decision of the Opposition Division to revoke European patent No. 0 389 061, which was granted in response to European patent application No. 90 200 664.2.

II. The decision under appeal was based on claims 1 to 47 as granted. It was held that the subject-matter of claim 1 as granted lacked an inventive step. Reference was made, inter alia, to the following prior art documents:

D1: US-A-4 446 117

D5: US-A-3 868 442

D6: US-A-3 681 013

D10: Chemistry and Industry of July 13, 1957,
pages 967-975,

D19: Light Metals 1981, pages 45-81.

III. With the statement of the grounds of appeal, the appellant (proprietor) filed on 24 October 1997 a new set of claims 1 to 47. Claim 1 thereof read as follows:

"Process for the production of aluminium hydroxide by digesting bauxite which contains alumina trihydrate together with alumina monohydrate, with an alkali solution in at least two digestion steps which are carried out at different temperatures and precipitating aluminium hydroxide from the digestion solution, characterised in that the process comprises:

a combining bauxite and alkali solution having a caustic concentration of at least 180 grammes per litre expressed as sodium carbonate and digesting partly the bauxite in the combined product in a first digestion step at relatively low temperature to yield a reaction product;

b separating the reaction product obtained in step a into a liquid phase and a solids/liquid slurry without discarding a part of the bauxite after the first digestion step;

c combining liquid phase and solids/liquid slurry obtained in step b and digesting the combined product in a second digestion step at relatively high temperature;

d releasing heat from the reaction product obtained in step c to preheat liquid phase obtained in step b;

e separating the reaction product after preheating in step d into a supersaturated sodium aluminate solution and undissolved material;

f precipitating aluminium hydroxide from the supersaturated sodium aluminate solution obtained in step e and separating aluminium hydroxide to leave a spent liquor; and

g recycling spent liquor obtained in step f to step a for use as alkali solution."

The process according to claim 1 now explicitly related to digesting bauxite containing aluminium trihydrate together with monohydrate, and required that no part of the bauxite after the first digestion step was discarded. It was argued that D5 related to the teachings of D6 and did not relate to the treatment of a bauxite containing monohydrate. D5 provided a solution for the problem of erosion in the indirect heat exchangers when treating high-silica bauxite. The

skilled person, faced with the corrosion problems in the high temperature digestion of monohydrate containing bauxite, would have had no reasons to contemplate the teachings of D5 and D6.

With a letter dated 21 August 2000, received by fax on 22 August 2000, a new set of claims 1 to 47 was submitted as an auxiliary request. Claim 1 thereof was further limited by indicating temperature ranges for the two digestion steps.

IV. The respondent (opponent) contested the appellant's arguments and maintained that the process according to claim 1 of the main request lacked an inventive step over D5 and D6 in combination with the common general knowledge in the art. Apart from the documents mentioned above, further reference was made to the following documents:

D11: Light Metals 1984, pages 307-324,

D18: Australian Corrosion Engineering, July 1974,
pages 13-16,

D22: Corrosion Engineering, third edition, 1986,
pages 1-92,

D24: Proceedings of Conference 28 of Australian
Corrosion Association, Nov. 1988, pages 5-8.1 to
5-8.11.

The respondent's arguments can be summarized as follows:

The process according to claim 1 of the main request

differed from the process disclosed in D5 only in the explicit requirements that the bauxite contained alumina trihydrate and alumina monohydrate, that granular material from the first digester was not discarded and that the spent liquor used as alkali solution had a caustic concentration of at least 180 g/l. The teaching of D5 was neither limited to the treatment of a specific bauxite nor to a process in which granular material from the first digester was discarded. The skilled person had no reason not to apply the process of D5 to a bauxite comprising monohydrate. Whether or not granular material was discarded depended upon the amount of inert material in the bauxite; if it contained much inert material such as quartz, the granular material from the first digester was discarded, if not, there was no reason to remove solid material between the first and second digester. D5 disclosed a caustic concentration of about 170 g/l. In view of the general tendency in the art to operate at higher caustic concentrations, the skilled person would consider slightly higher concentrations as now claimed. The process according to present claim 1 was, therefore, a normal design variation depending upon the quality of the bauxite. It was further obvious to reduce the free soda concentration in the second digester in order to reduce corrosion and it was well known that the presence of aluminate from the first digester would reduce the free caustic concentration in the liquor and thus its corrosivity. The problem faced by the proprietor was essentially an erosion-corrosion problem in the indirect heat exchangers. This problem was discussed in D5 and solved by dividing the slurry from the first digester into two portions, to heat exchange only the liquid portion in the indirect heat exchangers, to heat the solids containing portion by

direct contact with steam and to unite both streams before treating the combined streams in the second digester. The patent in suit solved the same problem in the same way.

- V. The appellant requested that the decision under appeal be set aside and that the patent be maintained with the claims 1 to 47 submitted with the letter dated 24 October 1997 as main request or be maintained with the claims 1 to 47 submitted with the letter dated 21 August 2000 as auxiliary request.

The respondent requested that the appeal be dismissed.

At the end of the oral proceedings, which were held on 20 September 2000, the decision was announced.

Reasons for the Decision

Main request

1. Claim 1 of the main request has been amended with respect to claim 1 as granted in that it now explicitly requires that the bauxite contains alumina trihydrate together with alumina monohydrate and no part of the bauxite is discarded after the first digestion step. These features are based on claim 2 and page 8, lines 7 to 13 of the original application respectively and restrict the protection conferred. The amendments, therefore, fulfil the requirements of Article 123 EPC, which was, in fact, not contested.
2. It is undisputed that the process according to claim 1 is new. It remains to be decided whether the subject-

matter of claim 1 involves an inventive step.

3. In the patent in suit a conventional two-stream process is discussed, in which a slurry of the bauxite in a part of the spent liquor, obtained in the wet grinding process, and the remainder of the spent liquor are preheated individually, mixed together and then subjected to extraction. Further according to the patent in suit two processes are in use for extracting alumina from bauxites containing an economically winnable amount of alumina monohydrate. In the first process the digestion of both mono- and trihydrate is carried out under digestion conditions suitable for monohydrate-containing ores. In the second process the trihydrate is first digested from the bauxite under digestion conditions suitable for trihydrate-containing ores and the resulting residue is then treated under digestion conditions suitable for monohydrate-containing ores. The disadvantages of these processes are the severe erosion and/or scaling which may occur in the case of a one stream process or the severe corrosion which may occur in the case of a two-stream process at high temperature and/or high free caustic concentration. It was an objective of the invention to avoid the occurrence of corrosion due to heating of strong alkali solutions (column 1, line 57 to column 3, line 34).

4. The respondent has not contested that the said two-stream process for treating monohydrate containing bauxite is state of the art, but has taken the view that the process disclosed in D5 comes closer to the subject-matter of present claim 1. The Board cannot share this view for the following reasons.

As already indicated above, D5 specifically relates to the treatment of high-silica bauxite and solves the erosion problems associated therewith (column 1, lines 49 to 55). With respect to the process conditions, continuous reference is made to D6 of the same inventor. In D6 reference is made to Darling Range bauxite (column 1, lines 23 to 32), which indisputably is a high-silica bauxite substantially free of monohydrate. The conventional high-temperature digesters are operated at a temperature of 290°F (143°C) which is typical for trihydrate bauxite and not suitable to extract monohydrate from bauxite (column 5, lines 4 to 7). Also the fact that D5 does not mention chemical corrosion or erosion/corrosion problems but only erosion problems, due to the presence of abrasive particles, is a clear indication that D5 only concerns the digestion of bauxite at moderate temperatures and caustic concentrations, not suitable to extract alumina from monohydrate, where chemical corrosion is not an issue. The respondent's argument that in D6 (column 1, lines 32 to 35) erosion problems are associated with high temperatures so that D5 and D6, although using different terminology relate to the same problem as the patent in suit, is not convincing. Said passage in D6 mentions that the erosion problem is particularly serious in the flash-cooling section where, after digestion at the high temperatures and pressures, the resulting slurry is flashed back to approximately atmospheric boiling point and pressure, and that the erosion is further accelerated due to the velocities encountered in the flash-cooling sections. In the Boards opinion said passage in D6 teaches the skilled person that the erosion problem is due to the high velocity of abrasive particles in the flash coolers, which are at a temperature below the digestion

temperature, ie below 143°C. It does not teach that erosion is due to the high temperature in the digester. To the skilled person it is evident that the erosion problems mentioned in D5 and D6 are essentially different from corrosion problems encountered in indirect, tubular, heat exchangers for preheating spent liquor to a temperature suitable for the extraction of monohydrate, which requires temperatures above 200°C (see D19, paragraph above Figure II). The Board, therefore, holds that D5 does not only relate to generically different subject-matter, it also does not deal with problems associated with the high temperatures needed to digest monohydrate. D5, therefore, is not a suitable starting point for the evaluation of inventive step of the subject matter of present claim 1; see Case Law of the Boards of Appeal, 3rd edition 1998, Chapter I-D, points 3.1 and 3.2, pages 111 to 113.

The process disclosed in D1 specifically relates to the digestion of monohydrate but is not closer to the subject-matter of claim 1 than the two-stream process discussed in the patent in suit, because the liquid phase from the low temperature digester is not directed to the high temperature digester.

5. In the absence of a more appropriate prior art document the definition of the problem to be solved by the invention should normally start from the problem described in the patent in suit. In the conventional two-stream process there is a high corrosion risk in the tubular heat exchangers where the spent liquor for the high temperature digester is heated. The temperature of the spent liquor from the tubular heat exchangers should be as high as possible to minimize

the amount of live steam to bring the spent liquor to its final temperature. In agreement with the patent in suit, the problem underlying the invention can be seen to be the reduction of corrosion in the spent liquor heaters for the high temperature digestion of monohydrate comprising bauxite. The patent in suit proposes to solve this problem by a process according to claim 1, whereby the spent liquor in a first digestion step is loaded with aluminate and then separated from the solids before it is heated to the temperature required for the second digestion step. In this way the free caustic concentration of the spent liquor is reduced. It is, therefore, credible that with the process according to claim 1 the corrosion by the alkaline solution in the high temperature heaters is reduced. This improvement was, in fact, not contested. The Board is therefore satisfied that the process according to present claim 1 actually solves the above-mentioned problem. It remains to be decided whether the claimed solution is obvious to a person skilled in the art.

6. The problem of corrosion by hot alkaline solutions has long been known in the art and is discussed in several of the cited documents.

In D10 some aspects of the stress corrosion of steel in caustic soda solutions is discussed. To reduce corrosion several steel protection methods are mentioned. Changing the process streams is not envisaged. In fact it is indicated that the precautionary measures [against caustic cracking] depend largely on considerable modifications of the corrosive, which is not possible in the Bayer process for the extraction of alumina from the Bauxite ore

(page 974, right hand column). The importance of sodium aluminate and minor impurities is said to be uncertain and it is advised to keep the uncombined soda concentration below 200 to 250 g/l Na₂O or the temperature below 85°C. Thus D10 does not point to the modification as now claimed.

7. D11 and D24, both concerning corrosion in alumina plants, mention a relationship between corrosion and aluminate content of the caustic liquor. According to D11, the presence of alumina in caustic solution permit operation at higher temperature than normally accepted in caustic plants (page 307). According to D24 the addition of sodium aluminate to the sodium hydroxide reduced the rate of corrosion (page 5-8.2). They contain, however, no measures how to change the process in order to increase the aluminate content of the spent liquor. An increase by directing the spent liquor through a first low temperature digestion step and separation of the liquor from the slurry leaving the first digester, as now claimed, is not suggested.
8. D19 comprises a literature survey of the major chemical reaction processes in digestion and slurry holding and a derivation of reactor design based on these studies as well as on plant tests. It does, however, not discuss the corrosion problem and does not disclose a specific plant structure. D19, therefore, cannot provide to the skilled person any incentive for the claimed solution of the corrosion problem.
9. D18 concerns corrosion evaluation and control in a caustic environment. As pointed out by the respondent, it comprises in the abstract on page 13 the general statement that corrosion and associated erosion takes

various forms within a caustic environment, due to changing temperatures, materials of construction, and cleaning and scale formation effects. It indicates that control of stress corrosion type failures should start at the design stage and continue throughout the manufacture and operation phases but does not provide concrete modifications of existing plants. The respondent referred to D18 and D22 to show that corrosion and erosion are associated problems, so that a skilled person faced with corrosion problems will also take into consideration documents relating to erosion such as D5 and D6. As already indicated above, the Board agrees that erosion enhances corrosion, but holds that in a situation where erosion does not play a role, such as in the tubular heaters in the high temperature two-stream process, the skilled person had no reason to take into consideration documents specifically related to erosion problems such as D5 and D6. The Board does not exclude that the skilled person, trying to solve a corrosion problem in the tubular heaters of the high temperature two-stream process, was aware of D5 and D6, but is of the opinion that, for the reasons set out above, he would not have had any incentive to use features disclosed therein for solving this particular corrosion problem.

10. D1 discloses a double digestion process for monohydrate containing bauxite to reduce iron contamination. In order to reduce erosion in the tubular heaters, it is proposed to discard a granular residue after the low temperature digestion step. Contrary to present claim 1, the liquid portion of the slurry leaving the low temperature digester is not used in the high temperature digestion step. Thus D1 clearly teaches away from the present process.

11. The other documents cited during the opposition and appeal proceedings do not disclose any more relevant subject-matter. There is thus no need to discuss them here in detail. In summary, therefore, none of the documents put forward by the respondent points to the solution of the corrosion problem as now claimed.

12. The Board cannot accept the respondent's argument that all the process features of claim 1 being known in the art, the skilled person must be free to adapt existing processes to the special circumstances required for a specific bauxite by introducing any such known features without being hindered by patent protection. In the Board's view, inventions frequently relate to new combinations of existing elements; only in rare cases are completely unknown elements introduced. Whether patent protection for a new combination of elements is justified depends only upon the question whether this new combination was obvious to the skilled person trying to solve an existing technical problem or not. It follows from the above that the new combination of process steps and conditions in the process according to present claim 1 was not obvious to a person skilled in the art. The Board, therefore, holds that for the subject-matter of present claim 1 patent protection is justified.

13. Confirmation of the considerations set out above is to be seen in the circumstance that, although the corrosion problems in the tubular heat exchangers operating at high temperatures were known at least since 1974 (D18) and that a part of the solution was known since 1975 (D5), the features disclosed in D5 have not been applied to solve these known problems in a high temperature two-stream process for digesting

bauxite containing monohydrate until 1989, the priority year of the patent in suit, despite extensive research during the intermediate period.

14. Claims 2 to 47 are dependent upon claim 1. The subject matter of these claims thus involves an inventive step for the same reasons as given above for claim 1. The main request is therefore allowable.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the first instance with the order to maintain the patent on the basis of claims 1 to 47 filed on 24 October 1997, a description to be adapted and Figures 1 and 2 as granted.

The Registrar:

The Chairman:

S. Hue

R. Spangenberg