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D E C I S I O N
of 5 December 2001

Case Number: T 0468/98 - 3.3.5

Application Number: 91904362.0

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Language of the proceedings: EN

Title of invention:

SILICATE PRODUCTS

Patentee:

INEOS Silicas Limited

Opponent:

Henkel Kommanditgesellschaft auf Aktien

Headword:

Granules/INEOS

Relevant legal provisions:

EPC Art. 56

Keyword:

"Inventive step (yes)"

Decisions cited:

-

Catchword:

-



Case Number: T 0468/98 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 5 December 2001

Appellant: INEOS Silicas Limited
(Proprietor of the patent) Bank Quay
Warrington WA5 1AB (GB)

Representative: Bakkum, Ruben Joseph
van Exter Polak Charlouis B.V.
P.O. Box 3241
NL-2280 GE Rijswijk (NL)

Respondent: Henkel
(Opponent) Kommanditgesellschaft auf Aktien
TFP/Patentabteilung
D-40191 Düsseldorf (DE)

Representative: -

Decision under appeal: Decision of the Opposition Division of the
European Patent Office posted 5 March 1998
revoking European patent No. 0 471 807 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. The appeal is from the decision of the opposition division revoking European patent No. 0 471 807. The decision was based on the granted claims as the main request and on the two sets of claims filed on 2 December 1997 as the first and the second auxiliary request respectively. Claim 1 of both auxiliary requests reads as follows:

"1. A method of treating a sodium silicate feedstock, comprising compacting said feedstock to provide a compacted sheet-like product which is subsequently broken up and sieved to provide compacted granules having a mole ratio $\text{SiO}_2/\text{Na}_2\text{O}$ of from about 1.5 to about 3.3:1, an average particle size in the range from about 0.3 mm to about 2.0 mm and a water content of 14-30%, wherein the granules are heated to a temperature above about 60°C in an agitated bed."

II. The opposition division held in its decision that the subject-matter of claim 1 as granted lacked novelty with respect to US-A-3 838 192 (D2). The subject-matter of claim 1 of the auxiliary requests did not involve an inventive step. The claimed process solved the problem of further improving the bulk density and reducing the attrition loss of compacted sodium silicate granules. The solution consisted in heat treating the compacted silicate granules under the conditions stated in claim 1. A discussion of what happened during the said heat treatment had revealed that it rounded up the sharp edges of the compacted granules and provided for continuously even surfaces of the granules. This was measurable in the form of increased density and reduced attrition loss of the final product. D2 explained in

column 7 the effect achieved by heat treating the sodium silicate particles in an agitated bed, namely rounding off the sharp edges of the particles, thereby obtaining continuously even surfaces. The heat treatment disclosed in D2 was performed, in close similarity with the claimed heat treatment, in the course of a post-processing step wherein granules were subjected to a finishing operation after having been densified (ie compacted). This disclosure rendered the claimed process obvious.

III. In the grounds of appeal the appellant contested that the claims of the 1st auxiliary request lacked an inventive step and made this request his main request. In a communication annexed to the summons to oral proceedings, the board drew the parties' attention to documents US-A-3 931 036 (hereinafter D4) and US-A-3 875 282 (D5) cited in the patent in suit. In reply thereto, the appellant submitted amended claims 1 to 5 on 31 October 2001 as the main and sole request and informed the board that he did not wish to be represented at the oral proceedings. Oral proceedings took place on 5 December 2001 in the absence of the appellant. Claim 1 filed on 31 October 2001 reads as follows:

"1 A method of treating a sodium silicate feedstock comprising compacting between rollers said feedstock to provide a compacted sheet-like product which is subsequently broken up and sieved to provide compacted granules having a mole ratio $\text{SiO}_2/\text{Na}_2\text{O}$ of from about 1.5 to 3.3:1, an average particle size in the range from about 0.3 mm to about 2.0 mm and a water content of 14-30%, wherein the compacted granules are heated to a temperature above about 60°C to 100°C in an agitated

bed."

- IV. The appellant presented inter alia the following arguments:

During pneumatic transport the compacted silicate granules appeared to severely break down, leading to unacceptable levels of fines of above 15%. For compacted silicate granules which already had a bulk density above 850 g/l, the invention solved the problem of improving the resistance to attrition of the granules. This improvement was illustrated in the patent in suit by the ball mill test. The value of about 5% indicated in Table 1 equated to a value above 15% fines after pneumatic transport. In view of the results in Table 1, which demonstrated that the heat treatment led to a reduction in the fines level, when measured by the ball mill test, of an order of magnitude after 10 minutes heat treatment, it was plausible that the level of fines after pneumatic transport would be reduced to an acceptable level. Neither D4 nor D2 mentioned anything at all about improving the attrition properties of the granules. D2 disclosed that the particles of silicate became rounded during the heat treatment process. However there was no suggestion that this change of shape in any way affected the attrition properties of the product. The skilled person could not be led to the claimed solution by the teaching of D2 because D2 did not address attrition in any way. The link between the disclosure of a rounding process in D2 and an improvement in attrition properties had been deduced with a full knowledge of the disclosure in the patent in suit. A skilled person would have viewed D2 and D4 as alternative ways to improve the bulk density of spray

dried silicates. He would not have assumed that the combination of the two processes would be capable of providing an improvement in attrition properties.

V. The respondent's arguments can be summarised as follows:

It clearly derived from D4 and D5 that parameters such as the compaction pressure, the thickness of the sheet, the water content of the spray-dried particles, or the optional heat treatment between the compaction step and the granulation step disclosed in D4 had an influence on the properties of the compacted granules. The examples of the patent did not sufficiently define the compacted sodium silicate granules used as the starting material. If compacted granules prepared by the embodiment of D4 including a heat treatment before granulation had been used as the starting material, the problem of attrition would not have existed. Therefore, it was doubtful that the technical problem of reducing attrition loss had been solved with all possible starting materials falling within the definition of claim 1. Furthermore, the examples only gave the results of the ball mill test and the appellant's affirmation that the value of 4.9% in the ball mill test equated to 15% fines after pneumatic transport was not derivable from the patent in suit. Even if the problem of reducing the attrition loss were considered to be solved, then it would have been obvious to the skilled person to perform the heat treatment disclosed in D4 after instead of before granulation. Furthermore it was self-evident that the heating step had to be carried out in an agitated bed for obtaining a uniform temperature distribution and for rounding off the granule edges. The skilled person faced with the

problem of improving the attrition properties would have thought about the possible cause for the high attrition of the granules and would have attributed it to the breakage or attrition of the sharp edges of the granules. According to the patent in suit rounding off the edges of the granules was a decisive aspect for minimizing the attrition loss. Once the skilled person had recognized this aspect of the problem, then the solution thereto was obvious in view of D2 which disclosed that the heat treatment reduced the surface irregularities and rounded off the sharp edges of the particles. Although D2 did not disclose a link between rounding off the sharp edges and reducing the attrition loss, it was logical for the skilled person that the attrition loss would be decreased by rounding off the granule edges. The appellant's arguments that the high attrition during pneumatic transport was due to the breakdown or disintegration of the silicate granules because of insufficient contact surface adhesion between the primary particles was not supported by the patent in suit. Further, it was inconsistent with the appellant's arguments that the compaction between rollers led to a relatively hard thin sheet. Even if the appellant's arguments about the breakdown or disintegration of the granules were accepted, then the skilled person would have been led by the teaching of D2 or D4 to the claimed solution. As D4 taught that the properties of the granules were improved by the heat treatment, the skilled person would have contemplated performing this heat treatment after the granulation step instead of before it, even if D4 did not specify which properties were improved. According to D2 the particles became somewhat plastic in nature at a temperature of 70°C. The skilled person would have inferred therefrom that in the case of primary

particles compressed by compaction between rollers, this would have resulted in an increase of the points of contact between the primary particles of the compacted granule and in a stronger bond of the primary particles together. Therefore he would have applied the heat treatment of D2 to the compacted granules of D4.

VII. The appellant requested that the decision under appeal be set aside and that the patent be maintained with claims 1 to 5 submitted on 31 October 2001. The respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.
2. The amendments introduced into claim 1 during the opposition and appeal procedures meet the requirements of Article 123(2) and (3) EPC. It is directly and unambiguously derivable from the original application (PCT publication) that the compacted sodium silicate granules used in the claimed method may be produced by compacting a sodium silicate feedstock between rollers to provide a sheet-like product which is subsequently broken up and sieved to provide the desired particle size range of compacted granules : see page 2, lines 9 to 14, and original claim 1. The heat treatment to a temperature up to 100°C is disclosed on page 4, lines 11 to 14, of the PCT application. The addition of the word "compacted" before "granules" in the heating step makes it clear that the compacted granules of sodium silicate are subjected to the heat treatment. Therefore the scope of protection of present claim 1 is restricted with respect to that of the granted claims.

3. The subject-matter of claim 1 is novel with respect to the cited documents. As this was no longer disputed by the respondent at the appeal stage, detailed reasons for this finding are not necessary.

4. D4 and D5 both disclose a method of treating a sodium silicate feedstock having a $\text{SiO}_2/\text{Na}_2\text{O}$ molar ratio within the range stated in claim 1, wherein the feedstock is compacted between rollers to provide a compacted sheet-like product which is subsequently broken up and sieved to provide compacted sodium silicate granules. The particle size of the sieved granules either falls within the range defined in claim 1 or overlaps therewith (see D5, claims 1 to 4 and 6; column 4, lines 32 to 43 and 63 to 66; column 5, lines 1 to 6). These documents illustrate how to obtain the kind of compacted sodium silicates the patent in suit aims to improve and both constitute an appropriate starting point for the assessment of inventive step. D4, which further exemplified compacted granules having a bulk density greater than 850 g/l and discloses the water content of the compacted granules, is considered to represent the closest prior art. The compacted alkali silicate granules obtained in D4 have bulk densities of 35-75 lbs/ft³ (about 560-1200 g/l), and a moisture content of 12-26%, preferably 18 to 22%. The particle size can consist of numerous particle size ranges such as 10-65 mesh (if Tyler screen size: 0.21-1.65 mm). The sodium silicate granules of Examples 5 and 6 have a $\text{SiO}_2/\text{Na}_2\text{O}$ ratio of 2.0/1.0 and 3.22/1.0 respectively, a moisture content of 15% and 17%, a bulk density of 72 lbs/ft³ (1153 g/l) and 66 lbs/ft³ (1057 g/l), 60% and 62% of the granules falling in the -10 +65 mesh range. The bulk density of the compacted granules prepared in Examples 2 to 4 is lower, ie from 46 lbs/ft³ to 48

lbs/ft³ (737 to 769 g/l) (see claim 1; column 2, line 63 to column 3, line 3; column 3, lines 35 to 49; Examples 2 to 7).

- 4.1 Starting from this prior art, the technical problem underlying the claimed process can be seen in the provision of a process for producing sodium silicate granules which have a high bulk density and an improved resistance to attrition, in particular during pneumatic transport.
- 4.2 It is proposed to solve this problem by the process as defined in claim 1, which differs essentially from the process of D4 in that the compacted sodium silicate particles are subsequently heated to a temperature in the range from about 60°C to 100°C in an agitated bed. It is shown in the examples of the patent in suit that the heat treated granules have a level of fines, measured by the ball mill test, which is from about 5 times to about 25 times lower than that of the starting compacted granules, depending on the heat treatment time. The appellant argued in the grounds of appeal that the value of 4.9% obtained by the ball mill test for the starting granules equated to an unacceptable value of above 15% fines after pneumatic transport. Although the level of fines of the heat treated granules after pneumatic transport is not given in the examples, it is plausible in view of the considerable reduction of the fines level obtained by the ball mill test that the level of fines after pneumatic transport has been reduced to an acceptable level.

At the oral proceedings the respondent expressed doubts that the technical problem was solved by the claimed process. He argued that the value of 4.9% obtained in

the ball mill test was meaningless since the amount of fines (<200 μm) of the starting compacted granules had not been measured. This argument is not convincing in view of the explanation on page 3 (lines 47 to 55) of the patent in suit as to how the ball mill friability was determined. As the sample of granules was sieved to remove oversize (>1200 μm) and undersize (<200 μm) before introduction into the ball mill, the percentage of fines formed during milling for 5 minutes can be determined by simple sieving. Knowledge of the amount of fines <200 μm before the test is not necessary since the fines were removed from the product before its introduction into the ball mill.

The respondent objected for the first time at the oral proceedings that the fine level above 15% after pneumatic transport was not stated in the patent in suit, thus implicitly questioning its correctness. However he provided no evidence that this value was incorrect. It is true that the patent in suit does not contain any value about the level of fines after pneumatic transport. However, European patent specification EP-B-0 471 049 which is from the same patentee as the patent in suit and has the same filing and priority dates, also concerns the treatment of compacted sodium silicate granules with the view to reducing their attrition loss during pneumatic transport. This patent specification, which was known to the respondent as he confirmed at the oral proceedings, discloses in Table II both the fine level obtained by the ball mill test and the fine level after pneumatic transport. According to Table II and Example 2, the starting compacted sodium silicate granules were obtained from Crosfield Chemicals Eindhoven Netherlands. They had a particle size similar to the

claimed one and a water content falling within the claimed range. The level of fines determined by the ball mill test was 0.8% below 125 µm and the level of fines after pneumatic transport was 11% below 180 µm. In the patent in suit, the compacted sodium silicate granules used as the starting product in the examples also come from Crosfield Chemicals Eindhoven Netherlands and the ball mill test gives a level of fines below 200 µm of 4.9%. In view of the level of fines disclosed in EP-B-0 471 049 for both the ball mill test and the pneumatic transport, the board considers it plausible that the value of 4.9% fines < 200 µm in the ball mill test of the patent in suit equated to a level of fines of above 15% during pneumatic transport. Therefore, in the absence of evidence to the contrary, the board sees no reason not to accept the appellant's arguments already presented in the grounds of appeal and not contested by the respondent until the oral proceedings.

The respondent's line of arguments that an improvement of the attrition resistance was not obtained with all kinds of compacted sodium silicate falling within the definition of claim 1, in particular with those compacted sodium silicate granules whose manufacture included the optional heat treatment before granulation disclosed in D4, cannot be accepted by the board for the following reasons. The respondent's affirmation that with these compacted granules the problem of attrition during pneumatic transport does not arise and thus no improvement of the attrition can be achieved, is not supported by any evidence, although the burden of proof rests on the respondent in this respect. Taking into account that the heat treatment before granulation disclosed in D4 is not performed in an agitated bed, that it is applied to a sheet or flakes,

ie to a product which is very different from the granulated product, and that the heated product is then crushed in a mill, it is not credible, in the absence of evidence, that this heat treatment would have the same effect on attrition as the claimed heat treatment and that the problem of attrition loss does not exist with those starting compacted granules. Concerning the starting granules used in the examples of the patent in suit, the board sees no reason not to accept the respondent's arguments that the compacted sodium silicate granules from Crosfield Chemicals of Eijsden Netherlands were obtained by compacting the feedstock **between rollers** since this is in agreement with the disclosure of the patent in suit, page 2, second paragraph, and with claim 1. As pointed out by the respondent at the oral proceedings, it appears that the properties of the starting compacted granules depend on certain parameters such as the compaction pressure, the thickness of the sheet or the water content of the spray-dried powder. However, it cannot be deduced therefrom that a reduction of the attrition loss would not be obtained with all kinds of compacted granules falling within the definition given in claim 1. The respondent has provided no evidence in support of his affirmation that the claimed heat treatment would not lead to an improvement of the attrition resistance for all kinds of compacted granules prepared as stated in claim 1, although the burden of proof lies on him. In the absence of such evidence and considering, on the other hand, that the examples of the patent in suit show a reduction of attrition loss resulting from the claimed heat treatment, the board cannot conclude that the technical problem is not solved over the whole breadth of claim 1.

For the preceding reasons the board considers it credible, in the absence of evidence to the contrary, that the technical problem has actually been solved by the claimed process.

5. The respondent's arguments that the teaching of D4 would have rendered the claimed solution obvious to the skilled person (see point VI above) are not convincing. The board observes that neither D4 nor D5 deals with the problem of attrition of the compacted granules prepared by the process disclosed therein, let alone with the attrition loss during pneumatic transport. These documents also do not disclose a heat treatment of the compacted granules. In the process of production of compacted alkali silicates according to D4, the compacted sheet is aged for 5 to 15 minutes before the granulation step. During this aging step the compacted material, either in the form of whole sheets or of flakes, can be subjected to heating and/ or cooling. During the aging step the compacted material is for example heated to a temperature of 65°C to 200°C for 3-8 minutes and then cooled to below 21°C in 1 to 10 minutes. Then the compacted material is granulated by crushing in various types of mills and the resulting granules are screened (see claims 1 and 6; column 1, lines 44 to 57; column 3, lines 19 to 39). The appellant's argument that the skilled person would have contemplated performing the heat treatment before instead of after the granulation step because D4 taught that this heat treatment resulted in improved properties of the granules cannot be accepted since D4 does not disclose an improvement of properties due to the said heat treatment. According to D4 the advantage obtained by heating the sheet or flakes after compaction is that the time needed for aging before

granulation is reduced : see column 3, lines 62 to 64. This effect which is in no way related to the attrition properties cannot suggest to the skilled person that performing a heat treatment of the compacted granules, ie a heat treatment after instead of before granulation, would have improved the attrition resistance during pneumatic transport. D5 which mentions no heat treatment and does not deal with the problem of attrition of the compacted granules likewise cannot point towards the claimed solution.

6. In the process for producing sodium polysilicate disclosed in D2, the spray-dried hollow microspheres are fractured and pulverized in the milling apparatus (9) in order to increase their bulk density and the pulverized particles are then passed into the rotary dryer (10) which serves to reduce the surface irregularities of the fractured particles and to form a product having continuously even surfaces. The temperature of the fractured particles in the rotary drum is preferably in the range of from 70 to 140°C. According to D2, at a temperature of about 70°C or higher the particles become somewhat plastic in nature. This characteristic is preferable in obtaining the desired reduction of the surface irregularities of the particles, ie obtaining a product having continuously even surfaces. It is further disclosed in Example 1 that the tumbling action of the rotary dryer has the effect of rounding off the sharp edges of the fractured particles so that the particles have continuously even surfaces (see claim 1, column 4, lines 45 to 70; column 7, lines 33 to 39; Figure 3). The heat treatment disclosed in D2 is thus applied to a spray-dried powder which has been fractured and densified in the milling apparatus (9), ie to a product which is very different

from the compacted granules as defined in claim 1.

The respondent's argument that the teaching of D2 about the effect of the heat treatment would have encouraged the skilled person to apply this treatment to the compacted granules of D4 is not convincing for the following reasons. Firstly it is not suggested in D2 that the presence of sharp edges on the densified particles, let alone on a different product such as the compacted granules of D4, might be responsible for the high attrition of above 15% fines during pneumatic transport since D2 does not deal in any way with the problem of attrition. Furthermore, the disclosure in D2 that the particles become somewhat plastic during the heat treatment is neither linked to nor associated with a possible improvement of attrition properties of the densified product of D2 or of compacted granules such as those of D4. D2 solely teaches that this plastic state is preferred for obtaining the desired reduction of the surface irregularities but does not disclose any positive effect on the attrition properties of compacted granules. According to the respondent, the skilled person would have inferred from the teaching of D2 that, in the case of primary particles compressed by compaction between rollers, the plastic nature of the heated particles would have resulted in an increase of the points of contact between the primary particles and thus in a stronger bond between these particles, leading to an improvement of the attrition properties. The board is not convinced by these arguments since this mechanism is neither described nor suggested in D2 and also not in D4 although the latter discloses a heat treatment of a compacted product in the form of flakes before granulation. Therefore, in the board's judgment, these considerations are based on the knowledge of the

patent in suit that the heat treatment of the compacted granules leads to an improvement of their attrition properties, and they rather represent possible explanations as to why the heat treatment results in the said improvement in the case of compacted granules.

The respondent further argued that the skilled person would immediately have realised that the breakage or attrition of the granule edges was responsible for the high attrition during pneumatic transport and that the claimed heat treatment was, thus, obvious in view of the teaching of D2 about the sharp edges becoming rounded during the heat treatment. These arguments cannot be accepted by the board for the following reasons. The respondent's assumption that breakage or attrition of the granule edges and not the breakdown of the granules is responsible for the high attrition is not confirmed by the explanations in the patent in suit. According to the patent in suit, the claimed heat treatment in an agitated bed improves the attrition properties of the compacted granules. The breakdown of the compacted granules (ie the crushing or destroying thereof: see Collins English dictionary, 3rd edition, and Oxford English dictionary) reduces as the temperature is raised and it becomes very low above about 75°C. In the attempt to explain the mechanism leading to both an increase of the bulk density and an improvement of the attrition properties, it is further referred on page 3 to the melting of the silicate in the water, granule shrinkage, edge rounding, bridging at the contact points between the primary particles and adherence of the fines to the larger particles (see lines 9 to 14). Moreover, the ball mill friability test, which is used according to the patent in suit for estimating the attrition behaviour, is said to measure

the breakdown of the granules under conditions representing high shear mixing (see page 3, lines 47 to 48). Therefore breakage or attrition of the granule edges is not presented in the patent in suit as the essential cause of the high attrition. In these circumstances, the board is not convinced that it was self-evident to the skilled person that the high attrition of above 15% during pneumatic transport is mainly due to the attrition or breakage of the granule edges. The appellant's argument that the compaction between rollers leads to a "relatively hard" thin sheet of silicate material is, contrary to the respondent's opinion, not inconsistent with the statement that breakdown of the granules occurs during pneumatic transport. A product which is hard may nevertheless break down under impact if it is brittle. Furthermore, the expression "relatively hard" has a relative meaning and can only be construed considering the whole method of preparation of these compacted granules. According to D4, the thin sheets or flakes produced in the compaction step are **easily granulated** by crushing in a mill (see column 3, lines 35 to 37). This is not incompatible with the fact that the granules may break down during pneumatic transport or handling.

The skilled person faced with the problem of improving the attrition properties of the compacted granules of D4 would have looked in the prior art concerning the same technical field, neighbouring technical fields or technical fields known to deal with the problem of attrition of granules to see whether he could find suggestions as to how this problem might be solved. However, as already indicated above none of the documents D4, D5 and D2 deals with the said problem or suggests that the heat treatment disclosed in D4 and D2

might improve the attrition properties of compacted granules. There is also no suggestion in these documents that the reduction of surface irregularities (rounding off the particle edges) which is achieved in D2 might solve the attrition problem encountered with compacted granules, ie a structurally different product. Therefore, the skilled person **not knowing the claimed solution** to this problem would have had no reasons to focus on the heat treatment of D2 which is not associated with any improvement of the attrition properties. Therefore, the board considers that the respondent's argumentation concerning inventive step is based on an ex post facto analysis.

7. The second document cited by the respondent in the notice of opposition (ie US-A-3 687 640) was no longer relied upon during the appeal procedure. The board is also of the opinion that this document which relates to a process for agglomerating alkali metal silicates and does not deal with the problem of attrition cannot point towards the claimed solution.
8. It follows from the above that the subject-matter of claim 1 meets the requirement of inventive step set out in Articles 52(1) and 56 EPC.

Claim 1 being allowable, the same applies to dependent claims 1 to 5, whose patentability is supported by that of claim 1.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the opposition division with the order to maintain the patent with the claims filed with the letter dated 31 October 2001 and a description to be adapted.

The Registrar:

The Chairman:

U. Bultmann

R. Spangenberg