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DECISION of 27 June 2001

0500813

| Case | Number: | Т | 0394/97 | - | 3.3.6 |
|------|---------|---|---------|---|-------|
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Application Number: 91903930.5

Publication Number:

IPC: D21C 9/10

Language of the proceedings: EN

Title of invention: Process for bleaching of ligno-cellulosic material

Patentee:

Eka Chemicals AB

Opponent:

(I) KVAERNER PULPING TECHNOLOGIES AB
(II) Degussa AG

Headword: Pulp bleaching/KVAERNER

Relevant legal provisions: EPC Art. 56

Keyword:

"Inventive step - no (diverging effects - prior art contains a guidance for combining various features in order to compensate for unavoidable brightness loss)"

Decisions cited:

-

Catchword:



Europäisches Patentamt European Patent Office Office européen des brevets

Beschwerdekammern

Boards of Appeal

Chambres de recours

Case Number: T 0394/97 - 3.3.6

D E C I S I O N of the Technical Board of Appeal 3.3.6 of 27 June 2001

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Appellant: (Opponent II) Degussa AG, Frankfurt - Zweigniederlassung Wolfgang -Postfach 13 45 D-63403 Hanau (DE)

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| Respondent: | | | | Eka Cł | nem: | icals AB | |
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Decision under appeal: Decision of the Opposition Division of the European Patent Office posted 12 February 1997 rejecting the opposition filed against European patent No. 0 500 813 pursuant to Article 102(2) EPC.

Composition of the Board:

Chairman: P. Krasa Members: G. Dischinger-Höppler C. Rennie-Smith

Summary of Facts and Submissions

I. This appeal is from the decision of the Opposition Division rejecting two oppositions and maintaining European patent No. 0 500 813 with the eight claims as granted, the only independent claim reading:

> "1. A process for reducing the amount of chloro-organic substance formed during delignification and bleaching of ligno-cellulosic pulp digested by chemical processes, characterised in that the pulp, in a first stage, is bleached with chlorine dioxide as the only chlorine-containing bleaching agent with a charge factor of up to 2.0 and at a pulp concentration of up to 15 percent by weight, and in a second stage, the pulp is treated with hydrogen peroxide at a pH above 7, and with a charge of at least 3.0 kg of hydrogen peroxide per ton of dry pulp, whereupon the treated pulp is bleached to the desired brightness with chlorine dioxide in one or more stages, wherein the ratio of the amount of chlorine dioxide charged in the prebleaching to the amount of chlorine dioxide charged in the final bleaching is within the range of from 1:1 to 1:5."

- II. The notices of opposition, based on lack of novelty and inventive step, relied inter alia on the following documents:
 - (1) B. Dillner et al., Tappi Proceedings, 1989 Pulping Conference, Book 1, pages 213-222;
 - (2) J. Basta et al., Tappi Proceedings, 1989 Pulping Conference, Book 2, pages 427-436; and

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- (3) H. U. Süss et al., Tappi Proceedings, 1989 Pulping Conference, Book 2, pages 527-537.
- III. The Opposition Division held that neither document (1) nor document (3) hinted at the claimed combination of features in order to reduce greatly the discharge of AOX whilst preserving the brightness and strength properties of the pulp.
- IV. In response to the Appellant's (Opponent I) appeal, the Respondent (Proprietor) filed a new set of claims in an auxiliary request, Claim 1 of which differs from granted Claim 1 in that the term "of up to 2.0" has been replaced by "within the range of from 0.6 to 1.8".
- V. Inventive step was the only point at issue during the oral proceedings held before the Board on 27 June 2001.
- VI. The Appellant, orally and in writing, submitted in essence
 - that a skilled person would use low multiple chlorination (= low charge factors or low kappa factors) in the first bleaching stage and peroxide in the extraction stage as suggested in document (1) in order to reduce the AOX level (adsorbable amount of organic halogen) in the effluent if bleaching efficiency is not important;
 - that document (2) also hinted at the lowering of the charge factor in order to decrease AOX and at the possibility to shift delignification work to an E-stage reinforced with oxidative chemicals.

VII. The Respondent's arguments can be summarised as

follows:

- The problem underlying the patent in suit was to reduce the AOX in the effluent while maintaining acceptable brightness and strength of the pulp.
- Documents (1) and (2) taught away from using charge factors below 2.0 in the first bleaching stage when using chlorine dioxide as the only chlorine-containing bleaching agent.
- Document (3), while relating to the same problem, contained, either alone or when combined with documents (1) or (2), no suggestion that this problem could be solved by the claimed ratios of the amounts of chlorine dioxide charged in the prebleaching and in the final bleaching in combination with the high charge of hydrogen peroxide in the first extraction stage.
- VIII. Opponent II as a party as of right commented that the closest prior art was represented by document (3) which already suggested a solution to the problem given in the patent in suit. This solution differed from the claimed subject-matter only in that a lower amount of hydrogen peroxide was used in the E stage. Use of higher amounts of hydrogen peroxide was, however, proposed in document (1).
- IX. The Appellant requested that the decision under appeal be set aside and that the patent be revoked.

The Respondent requested that the appeal be dismissed or alternatively that the patent be maintained according to the auxiliary request filed during the oral proceedings.

Reasons for the Decision

Main Request

Lack of novelty being no longer in dispute, the only issue to be decided is whether or not the claimed process is based on an inventive step.

- 1. Technical background
- 1.1 The patent in suit relates to a process for bleaching of ligno-cellulosic pulps wherein formation of chloroorganic substances which can be measured as AOX is reduced whilst final brightness and strength properties of the pulp are preserved (page 2, lines 3 to 4 and lines 26 to 27).
- 1.2 According to the patent in suit, it is known that AOX can be reduced by using chlorine dioxide instead of molecular chlorine as the bleaching agent and/or by reducing the charge factor (the amount of chlorine containing bleaching agent, calculated as active chlorine, in the first bleaching step). A further possibility consists in a pretreatment with oxygen to decrease the lignin content of the pulp which corresponds to a reduced kappa number (page 3, lines 5 to 10 in combination with page 2, lines 16 to 20). However, use of chlorine-dioxide alone for bleaching and lowering the charge factor makes it difficult to achieve sufficient final brightness (page 3, lines 11 to 13).

- 1.3 Hence, the patent seeks to provide a process with decreased discharge of AOX by using chlorine dioxide as the only bleaching agent and in reduced amounts in the prebleaching stage in a manner providing an acceptable final brightness (page 4, lines 4 to 7). From the description of the patent in suit it can be inferred that an acceptable AOX would be below 1 kg/ton of pulp (page 5, lines 56 to 58) and that a brightness of 89.5% ISO would be sufficient (page 5, lines 13 to 14). The Respondent nevertheless stated during the oral proceedings that the claimed subject-matter should be interpreted as also covering tests 2 to 4 of Table III. The final brightness of the pulps obtained may accordingly be as low as 86.3% ISO (see test 2).
- 1.4 As concerns the strength of the pulp expressed as its viscosity, it can be deduced from Example 2 that at least a decrease in viscosity from 1040 dm³/kg, based on the oxygen pretreated pulp (page 5, lines 18 to 19), to a final viscosity of about 920 dm³/kg is deemed not to affect adversely the strength properties of the pulp and thus be acceptable (page 6, lines 40 to 42). On the other hand, no figures are given for the several tests in Example 3 of the patent in suit including those allegedly embraced within the claimed subject-matter. Therefore, no evidence exists in the patent demonstrating a particularly reduced viscosity loss.

2. Closest prior art

2.1 Document (3) is a scientific article which, in general, relates to the problem of reducing AOX (page 527, right-hand column, first paragraph) and is therefore a suitable starting point for assessing inventive step. 2.2 Section 6 is specifically concerned with the "Minimization of halogenated compounds in the effluent" (pages 533 to 534). In this section, a comparison is made between oxygen pretreated bleaching sequences wherein 50% and 100% respectively of the chlorine content in the first bleaching stage was substituted by chlorine dioxide. As can be seen from Table 2 (page 534), an AOX discharge of below 1 kg/t pulp (0.81 kg/t) is achieved whilst obtaining a high brightness of above 90% ISO (90.2) and a viscosity of above 19 mPas (19.1 mPas) by using chlorine dioxide as the only bleaching agent. This results from an O-D-EOP-D-P-D-P-sequence, a so-called "long sequence" which comprises two D stages (here D-P-D-P) in the final bleaching sequence and a peroxide-reinforced alkaline extraction stage EOP in the prebleaching sequence (here D-EOP). The amount of chlorine dioxide used in the prebleaching stage is given as a kappa factor of 0.2 and, depending on the initial kappa number, as being equivalent to a content of 4% active chlorine in the pulp. The kappa factor of 0.2 unarguably corresponds to a charge factor of 2.0 according to the definition given in the patent in suit (page 2, line 57 to page 3, line 2). By contrast, the chlorine dioxide used in the final bleaching stage amounts to a total of only 2.3% (1.7% + 0.6%) calculated as active chlorine. The alkaline extraction stage was reinforced with 0.25% H₂O₂ (i.e. 2.5 kg hydrogen peroxide per ton of pulp) and the whole bleaching sequence was carried out at a consistency of 10% (page 537, lines 13 to 22).

3. Technical problem

3.1 It follows from the above that document (3) already contains a suggestion of how to achieve the desired

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AOX level of below 1.0 kg/ton of pulp in the effluent at an acceptable final brightness.

- 3.2 Concerning the strength of the pulp, document (3) is silent about any particular value for the viscosity of the oxygen-pretreated pulp before the bleaching. Therefore, a decrease in viscosity due to the bleaching treatment cannot be estimated. Moreover, the viscosity values in Table 2 of document (3) after the bleaching are given in mPas and are not comparable with those expressed in dm³/kg in the patent in suit. However, as was agreed by the parties, a certain preservation of the strength properties of the pulp is always a prerequisite if a papermaking process is to be useful. So this would be the case in the document (3) process.
- 3.3 As a consequence, the technical problem to be solved as against document (3) amounts to providing an alternative bleaching process with an AOX discharge of below 1 kg per ton of pulp at acceptable final brightness without unduly worsening the strength properties of the pulp.
- 4. Solution of the problem
- 4.1 Oxygen pretreatment is not precluded from the claimed bleaching process, nor is the long-sequence bleaching. This is corroborated by the fact that all examples in the patent in suit are made with oxygen-delignified sulphate pulp (page 5, lines 18 to 19) and two D stages in the final bleaching sequence. Therefore, the solution to this problem as proposed by Claim 1 of the patent in suit consists in performing trial 2 of Table 2 in document (3) such that

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the amount of chlorine dioxide charged in the final bleaching stage is equal to or up to five times the amount used in the prebleaching stage (feature (a)) and that

 at least 3.0 kg of hydrogen peroxide per ton of pulp is used in the alkaline extraction stage (feature (b)).

- 4.2 It is evident from the example of the patent in suit that under such conditions the AOX discharge can be decreased below 1 kg/ton of pulp whilst retaining the brightness and strength properties at acceptable levels.
- 5. It remains to be decided whether, in view of the available prior art documents, it was obvious for someone skilled in the art to solve this problem by the means claimed.
- 5.1 Document (3) teaches that low kappa factor chlorination decreases the amount of AOX discharged, but results in insufficient delignification (page 527, left-hand column, second paragraph). As is evident from Figures 6 and 7 in document (3) for oxygen-predelignified pulp (and from Figures 3 and 5 for pulps not so treated), a lowering of the kappa factor, i.e. the charge factor, generally results in diverging effects as regards AOX discharge and delignification of the pulp. Figure 6 shows that bleaching of pulp which has been predelignified with oxygen to a kappa number of 20.0, using chlorine dioxide as the only bleaching agent at a kappa factor of between 0.1 and 0.25, gives AOX values no higher than 0.5 kg/ton and that the AOX discharge decreases as the kappa factor decreases. This effect

occurs, however, at the expense of delignification. As can be seen from Figure 7, the kappa number - which corresponds to the degree of delignification increases as the kappa factor decreases. Thus, document (3) already imparts the information that a compromise has to be made between optimum delignification and optimum AOX discharge. This is corroborated by document (2), where Figure 2 shows how final brightness and AOX discharge depend on each other, and where Figure 5 illustrates the relationship between charge factor and kappa number for several bleaching conditions.

- 5.2 However, document (3) also offers a means which compensates for lower degrees of delignification, namely by an enforcement of the extraction stage with hydrogen peroxide. Very low levels of AOX are said to result from a combination of oxygen pre-delignification followed by chlorine dioxide and hydrogen peroxide treatment (page 527, left-hand column, paragraphs 3 and 4). Therefore, contrary to the Respondent's opinion, the authors of document (3) did find a compromise in trial 2 of Table 2 which produces an optimum combination of the requirements of low AOX discharge, high brightness and sufficient pulp strength via the oxidative reinforcement of the extraction stage with hydrogen peroxide (see also page 533, right-hand column, lines 5 to 9 from the bottom).
- 5.3 Thus, document (3) already suggested to the skilled person that he had to add hydrogen peroxide in the extraction stage if he wanted to compensate for the unavoidable brightness loss which pulp undergoes in a compromise between an AOX reduction and delignification.

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- 5.4 As to the hydrogen peroxide amount of at least 3 kg per ton of pulp, it is to be noted that no particular relevance can be attached to this value, i.e. to the above feature (b) (see 4.1 above). Therefore, this value appears to result from routine trials not requiring any inventive activity. Moreover, the skilled person could also find concrete guidance for this in document (1).
- 5.5 That citation also discloses the beneficial effect on the brightness of the pulp of peroxide addition in the extraction stage (page 217, left-hand column). Figure 11 shows that brightness is increased with increasing amounts of hydrogen peroxide added in the extraction stage and Figure 12 shows that viscosity is not adversely changed up to a level of about 4 kg hydrogen peroxide per ton of pulp. Higher amounts are said to be negative for the pulp viscosity (page 218, right-hand column, lines 5 to 12). As is shown in Figure 12, even higher amounts of peroxide can nevertheless be considered if some viscosity loss is accepted. Thus, document (1) provides an incentive for feature (b), i.e. to use higher amounts of hydrogen peroxide (3 kg/ton and above) in the extraction stage to compensate for any poor delignification in the first bleaching stage.
- 5.6 As to feature (a), document (1), whilst not preferring it, also considers low active chlorine charges in the first bleaching stage even when chlorine dioxide is the only bleaching agent (Figure 5; the term "multiple" used therein is equivalent to the kappa factor of document (3)). Multiples below 0.20 are said to be disadvantageous because of poor bleaching efficiency, i.e. the overall consumption of active chlorine for a

given brightness, and because of decreased viscosities. In addition, a brightness of 90% ISO is said to be difficult to achieve (page 215, right-hand column, line 5 to page 216, left-hand column, line 5). However, if - as in the present case - maximum brightness and viscosities are not in issue, these disadvantages cannot be considered as a prejudice or warning against using low multiple chlorination for less ambitious purposes; it is to be borne in mind that a brightness of the pulp as low as 86.3% ISO is acceptable according to the patent in suit (see 1.3 above).

- 5.7 For the same reason, and contrary to the Respondent's opinion, document (2) does not teach away from the claimed subject-matter. Document (2) is primarily concerned with the requirements for obtaining 90% ISO brightness (page 429, right-hand column, lines 1 to 9). This does, however, not distract a skilled person prepared to accept lower brightness from the general teaching that lowering the charge factor in the prebleaching step would be an effective way of decreasing the AOX level in the effluent and that any insufficient delignification in the first bleaching stage could be compensated for by reinforcing the extraction stage with oxidative chemicals (page 431, left-hand column, last paragraph and right-hand column, first paragraph). This is exactly the route followed in the patent in suit which, thus, does not require inventive skills.
- 5.8 Further, Figure 5 of document (1) offers low multiple chlorination as an option for bleaching pulp predelignified with oxygen to a kappa number of 13.2 via a D-EO-D-D bleaching sequence. If, for example a brightness of 89% ISO was aimed at, one option consists

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in using a chlorination multiple of 0.14 (corresponding to a charge factor of 1.4) in the first bleaching stage at a total of about 54 kg of active chlorine per ton of pulp. Since a chlorination multiple of 0.14 corresponds to 18 kg/ton of active chlorine (for kappa number 13.2), the Board agrees with the Appellant's undisproved explanation that in the final bleaching stages 36 kg/ton (54 minus 18) must be used for bleaching to the desired brightness of 89% ISO, thereby arriving at the conditions of feature (a) (see 4.1 above). According to Figure 6, a final viscosity of about 870 dm³/kg can be expected for this trial, corresponding to an acceptable viscosity drop of around 100 dm³/kg (from 966 to 870).

- 6. The Board agrees with the Respondent's submission that, when evaluating inventive step, it is the whole combination of features which has to be investigated in its entirety and not single components of the combination picked out as favourable features from various documents. It is correct that it is the inventiveness (or obviousness) of the combination (here, of process features) which has to be investigated, i.e. that of the claimed process. However, the decisive point is whether or not the state of the art offered the skilled person sufficient guidance to bring various features together to achieve a certain result, i.e. to solve the existing technical problem. In the present case, such guidance was to hand as explained in points 3 to 5 above and, by following it, the skilled person would plainly have arrived at the process of Claim 1.
- 7. The Board, therefore, concludes that, for the purpose of providing an alternative to the process according to

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document (3), the skilled person considering the consequences of using a low charge factor in the first bleaching stage would readily have adapted the amounts of chlorine dioxide in the final bleaching stages and the amount of hydrogen peroxide in the extraction stage as recommended in documents (1) and (2) with the expectation of getting results as good as those in Figure 2 of document (3). He would thereby arrive at the subject-matter of Claim 1 in an obvious manner.

For these reasons, the Board finds that the process of Claim 1 does not comply with the requirements of Articles 52(1) and 56 EPC; the main request must fail.

Auxiliary request

- 8. No objections under Articles 84 and 123 EPC arise from the restriction of the subject-matter of Claim 1 with respect to the charge factor used in accordance with Claim 5 as originally filed (corresponding to Claim 4 as granted).
- 9. This restriction does not, however, add any inventive feature to Claim 1 of the main request since charge factors falling within the range considered in Claim 1 of the auxiliary request are already considered in document (1).

10. The same conclusions as drawn for Claim 1 of the main request (see point 7 above) therefore apply mutatis mutandis to Claim 1 of the auxiliary request.

Order

For these reasons it is decided that:

- 1. The decision under appeal is set aside.
- 2. The patent is revoked.

The Registrar:

The Chairman:

G. Rauh

P. Krasa