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**D E C I S I O N**  
**of 11 July 2002**

**Case Number:** T 0715/99 - 3.3.6

**Application Number:** 94901143.1

**Publication Number:** 0670928

**IPC:** D21C 9/10

**Language of the proceedings:** EN

**Title of invention:**

Process for delignification of lignocellulose-containing pulp

**Patentee:**

Eka Chemicals AB

**Opponents:**

Kvaerner Pulping AB  
SOLVAY INTEROX (Société Anonyme)  
Kemira Chemicals OY

**Headword:**

Delignification/EKA

**Relevant legal provisions:**

EPC Art. 56, 114(2)

**Keyword:**

"Late-filed document - not admitted"  
"Inventive step - yes: no incentive in the prior art for the  
claimed solution of the technical problem"

**Decisions cited:**

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**Catchword:**

-





Case Number: T 0715/99 - 3.3.6

**D E C I S I O N**  
of the Technical Board of Appeal 3.3.6  
of 11 July 2002

**Appellant:** Kemira Chemicals OY  
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**Representative:** -

**Decision under appeal:** Interlocutory decision of the Opposition Division  
of the European Patent Office posted 21 May 1999  
concerning maintenance of European patent  
No. 0 670 928 in amended form.

**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 interlocutory decision of the Opposition Division to maintain in amended form European patent No. 0 670 928. Claim 1 as maintained reads:

"1. A process for delignifying and bleaching lignocellulose-containing pulp, characterised in that

the pulp is delignified with an organic peracid or salts thereof,

whereafter the pulp is treated with a complexing agent in a separate stage and washed,

and subsequently bleached with a chlorine-free bleaching agent comprising at least one of a peroxide-containing compound, ozone or sodium dithionite, or optional sequence or mixtures thereof."

II. Three notices of opposition based on lack of novelty and inventive step (Articles 100(a), 54 and 56 EPC) cited inter alia the following documents:

(1) JP-57-21591 (and its English translation);

(2) EP-A-0 402 335;

(3) EP-A-0 480 469 and

(5) TAPPI, 1992 Pulping Conference, Book 3, page 1219 to page 1230.

- III. In its decision, the Opposition Division found that the claims as amended complied with the requirements of Articles 123(2) and (3), 84, 54 and 56 EPC. Concerning inventive step, the Opposition Division held that, in order to improve the brightness of the pulp as against the Q-W-P sequence of document (2), it was not obvious from the prior art, in particular document (1), to perform a PA-stage in advance of this sequence.
- IV. An appeal was filed only by Opponent III (Appellant). However, during the appeal proceedings, Opponent I filed document (9) (= US-A-3 876 246) and submitted in writing that the claimed subject-matter was not inventive over a combination of this document with document (2).
- V. Oral proceedings before the Board of Appeal were held on 11 July 2002 in the absence of the Respondent (Proprietor) and Opponents I and II as parties as of right as announced by letters of 28 May 2002, 4 January 2002 and 28 June 2002 respectively.
- VI. The Appellant submitted that the claimed subject-matter was not based on an inventive step for the following reasons:
- It was known from document (2) that a peroxide (P) stage was more effective if those metals which are most detrimental to the decomposition of hydrogen peroxide such as manganese (Mn) and iron (Fe) were considerably reduced by a separate preceding treatment with a complexing agent (Q stage) instead of a simultaneous treatment with Q and P.

- In order to improve the process of document (2), a skilled person would be guided simply to add an initial peracetic acid (PA) stage since documents (1) and (3) disclosed that PA was a good delignification agent.
  
- Whilst it was known from document (3) that in a PA-W-P sequence pulp can be treated by a chelating agent either in an initial separate stage or during the PA stage, the number and location of the Q stage(s) would be chosen with a view to optimization. Since the process of document (3), if carried out in the presence of a complexing agent in both the PA and P stages (Q/PA-W-Q/P sequence), resulted in substantial loss in viscosity between PA and P, it was obvious from document (2) to place the Q stage separately before the P stage.
  
- According to the patent in suit, the claimed subject-matter was not limited to the basic sequence PA-Q-W-P recited in Claim 1 but included preceding stages for treatment of the pulp in the presence of complexing agents.
  
- Document (5) suggested that peracetic acid was not sensitive to metal catalysed decomposition if unreacted peroxide was absent.

VII. The Respondent's written submissions were in summary that

- pretreating the pulp with a separate Q stage gave the best results for the PA/P bleaching of document (1); thus, there was no incentive for a

skilled person to place a PA stage before the Q stage in the Q-W-P sequence of document (2);

- the Q-W-P sequence of document (2) did not give rise to any modification of the Q-W-PA/P sequence of document (1) by placing the separate Q stage between PA and P; nor did document (3) or any of the other cited documents contain any hint of carrying out a separate Q stage between PA and P;
- document (9) was not more relevant than the other cited prior art and should be disregarded under Article 114(2) EPC;
- as was apparent from the experimental data filed during the opposition proceedings, the change of sequence according to the patent in suit surprisingly resulted in improved brightness and viscosity.

VIII. The Appellant and Opponent I in writing requested that the decision under appeal be set aside and that the patent be revoked.

The Respondent requested in writing that the appeal be dismissed and that the patent be maintained as amended.

The other party (Opponent II) made no request.

## **Reasons for the Decision**

### 1. *Late filed document*

1.1 About six weeks after the summons to oral proceedings,



the non-appealing Opponent I sought for the first time to rely on document (9). The only reason for doing so given in its letter dated 7 January 2002 is "to further prove the state of the art in bleaching". In particular, it stated that document (9) showed the advantages of P-PA-P, P-W-PA-W-P and PA-W-P-W-PA-W-P sequences and the explicit knowledge of the 'heavy metal problem' in respect of decomposition of the per compounds as well as the solution to this problem by adding complex builders.

1.2 The Boards of Appeal at the EPO often exercise their discretion under Article 114(2) EPC to admit late-filed evidence into the proceedings provided, inter alia that it is prima facie more relevant with regard to the claimed invention than the citations already on file, and that it might change the outcome of the decision to be taken by the Board.

1.3 In the present case, however, all the information mentioned above under 1.1 and being of relevance to the claimed process was already on file. It can be derived from document (3) in particular, which discloses the combination in one bleaching sequence of PA and P stages, both in the presence of complexing agents in order to prevent catalytic decomposition of the peroxy compounds by transition metal ions, and with intermediate washing recommended (Examples 7 and 9 and page 7, lines 18 to 29).

1.4 The Board holds, therefore, that document (9) should not be taken into consideration as not being prima facie technically more relevant than the documents already on file (Article 114(2) EPC).

2. The Board confirms the findings of the Opposition Division that the amendments made to the claims during the opposition proceedings comply with the requirements of Articles 84, 123(2) and (3) EPC, and that the subject-matter of these claims is novel over the cited prior art (Article 54 EPC). This not having been contested by any party during the appeal proceedings, no further comment on this matter is necessary.

3. The only issue to be decided is, therefore, whether or not the claimed subject-matter is based on an inventive step.

### 3.1 Technical background

The patent in suit is concerned with chlorine-free processes for delignifying and bleaching lignocellulose-containing pulp to produce fully bleached pulp with unaltered strength properties in a reasonable number of stages and with a reasonable consumption of bleaching agent (page 2, lines 31 to 34) and suggests to apply a PA-Q-W-P sequence at an optional point within a bleaching process, preferably immediately after preceding oxygen delignification (page 4, lines 40 to 42 and Examples).

### 3.2 Closest prior art

In the oral proceedings, the Appellant based its arguments on document (2), in particular Example 4, as the closest prior art, but as an alternative also used Example 7 of document (3) as a starting point for the evaluation of inventive step.

Both documents relate to chlorine-free bleaching of

pulp to high brightness and a low kappa number (indicating high delignification) with low viscosity loss (document (2), page 5, lines 10 to 14 in combination with page 3, lines 9 to 26; in document (3), page 4, lines 34 to 35 and 44 to 46) and, thus, both have essentially the same object as the patent in suit. Moreover, both documents use peracetic acid and/or peroxide for delignification and bleaching, preferably in combination with a complexing agent (document (2), page 3, lines 31 to 37; document (3), page 7, lines 9 to 24).

Since, further, both documents are mentioned in the patent in suit as background art, the Board holds that they are equally suitable as a starting point for the assessment of inventive step.

### 3.3 Technical problem solved in view of document (2)

Concerning document (2), it is stated in the patent in suit that pretreatment of a chemical pulp with a complexing agent directly after digestion or oxygen delignification makes a subsequent P stage more efficient (page 2, lines 22 to 23).

In detail, document (2) describes treating the pulp in a Q-P sequence, in particular a Q-W-P sequence, preferably after an oxygen stage in order to exclude charges of chlorine or chlorine dioxide from the bleaching while still providing good delignification and bleaching results (page 3, lines 31 to 37, page 3, line 57 to page 4, line 5 and page 4, lines 39 to 44). The effect of the Q stage is attributed to the reduction of metals, above all manganese contained in the pulp which is especially unfavourable to the P

stage, whilst essentially preserving the magnesium (Mg) content which is said to have a positive effect on the P stage (page 4, line 49 to page 5, line 3 and Example 4). The effect of carrying out a washing between the Q and P stages as compared to no intermediate washing is illustrated in Example 4 and consists in improved delignification (lower kappa number) at lower peroxide consumption and higher strength (higher viscosity).

The technical problem credibly solved by the claimed subject-matter can be derived from Example 3 of the patent in suit (see Table III) where it is shown that in comparison with a Q-W-P sequence, a preceding PA stage in accordance with the claimed sequence provides considerably improved brightness and kappa number with only a small decrease in viscosity.

### 3.4 Inventive step

It remains to be decided whether, in view of the available prior art documents, it would have been obvious to someone skilled in the art to solve that problem by the means claimed.

- 3.4.1 The Appellant argued that it was essential for the assessment of inventive step to consider that the claimed process was not limited to a particular bleaching sequence or chemicals to be used, but included further bleaching stages, in particular before and after the PA-Q-W-P sequence of Claim 1, such as preceding stages where complexing agents were added and therefore present before or during the initial PA stage. Reference was made in this respect to page 3, lines 39 to 46 of the patent in suit, according to

which it was possible to recycle for washing the spent liquors from the bleaching and treatment stages, thereby enabling a closed system in the mill. Since the amount of complexing agent used in the Q stage of up to 10 kg/ton pulp corresponded to a high surplus in respect of the heavy metal ions to be removed, the patent in suit covered recycling of unreacted complexing agent prior to the PA stage and, hence, a Q-PA-Q-W-P sequence.

In fact, the patent in suit states that the claimed process can be carried out **at an optional point** in the bleaching sequence. However, this does not, in the Board's opinion, necessarily mean **any** point, including a point after a preceding Q stage. Particular mention is made of a point immediately after making the pulp and after an initial oxygen stage (page 4, lines 40 to 42). All examples concern application of the claimed bleaching sequence to such oxygen-delignified pulp. Nothing in the patent in suit gives a hint to perform Q, PA or P stages before the claimed PA-Q-W-P sequence. Also the passage referred to by the Applicant does not suggest that any complexing agent remaining after a Q stage should be reintroduced for the purpose of washing before the PA stage, let alone under conditions suitable for complex formation with manganese. It merely says that due to its neutral pH, such liquor is useful for washing elsewhere in the mill. The Board, therefore, does not accept the Appellant's interpretation of the subject-matter claimed in the patent in suit, but holds that in the light of the description as a whole, the claimed subject-matter does not cover embodiments with a Q stage before the first PA stage.

3.4.2 The Appellant further argued that there was a hint in document (2) to increase delignification by two successive oxygen stages, i.e. by a second oxygen stage immediately after the initial oxygen delignification which had, however, turned out to be inefficient. Reference was made in this respect to page 4, lines 33 to 48. Since documents (1) and (3) already recommended peracetic acid for delignification, it was obvious for the skilled person to perform a PA stage instead of such a second oxygen stage, thus arriving at a O-PA-Q-W-P sequence which was within the terms of Claim 1.

3.4.3 Indeed, document (1) describes peracetic acid as having "superior delignification effect and bleaching power". Acknowledging that use of peracetic acid had already been proposed in the art in O-PA-P and P-PA-P sequences, document (1) is, however, concerned with the particular problem that peracetic acid is too expensive to be an economically relevant bleaching agent (page 3, paragraphs 1, 3 and 4). In order to reduce peracetic acid costs and in the interest of economy, document (1) suggests directly using the hydrogen peroxide left after the PA stage by activation with alkali in a combined PA/P stage without intermediate washing (page 3, paragraphs 5 and 6 and page 4, first full paragraph) and to prevent or limit decomposition of the **peracetic acid as well as of the hydrogen peroxide** in the presence heavy metal catalysts by treating the pulp during or before the combined PA/P stage with a chelating agent (page 3, last paragraph to page 4, line 3, page 4, fifth full paragraph).

It is evident from the examples given in document (1) that the effect, in terms of final brightness of the

pulp and reagent consumption in the process, of a combined Q/PA/P treatment where the chelating agent is added during the PA/P stage (Example 1) can be improved by a preceding Q stage (Example 2). Consequently, document (1) teaches to improve the prior art by carrying out a combined PA/P stage, preferably in the presence of a chelating agent as Q/PA/P stage and most preferably with an additional preceding Q stage, giving a Q-Q/PA/P sequence.

Considering the bearing on viscosity and kappa number of the final pulp of the intermediate washing in the Q-W-P sequence of document (2) (Example 4), any combination of these documents would, therefore, result in a Q-W-Q/PA/P sequence, the more so as an intermediate washing after the Q stage (as defined by dewatering the pulp from a concentration of 8% to 25% and a then necessary dilution to 15% pulp concentration; see also Table 3) is also performed in Example 2 of document (1). Even if one was, for the sake of argument, to assume that a skilled person would consider doing without the advantages of a combined PA/P stage and perform separate PA and P stages, documents (1) and (2) would not give him any incentive to perform a Q stage between PA and P, since he would expect, from the teaching on page 4 of document (1), the peracetic acid to decompose in the absence of a chelating agent.

- 3.4.4 Document (3) also describes using peracetic acid in delignification and bleaching of pulps (page 6, lines 20 to 23) and suggests in general a process for oxygen-delignification of pulp and in particular its combination with a preceding or subsequent treatment with a peroxy compound which is **either** peracetic acid

or hydrogen peroxide (page 6, lines 20 to 26 and Claims 4, 5, 14, 15 and 17).

According to the patent in suit, document (3) teaches that using sequences with both PA and P stages before or after oxygen delignification would result in significant loss of viscosity (page 2, lines 24 to 27).

Such a combination of PA and P stages is indeed mentioned in Example 7 (run 5) of document(3) as a O-Q/PA-Q/P sequence where both the PA and P stages are carried out in the presence of a complexing agent with intermediate washing after peroxy compound treatment in accordance with the general description (page 7, lines 28 to 30). As becomes evident from a comparison with Example 6, run 4, the addition of a Q/P stage after Q/PA increases the final brightness of the pulp from 64.7 ISO to 76.5 ISO and decreases the kappa number from 4.0 to a value too low to be measured, however at the expense of strength as expressed by a reduction in viscosity from 22.3 cps to 14.8 cps. No such viscosity loss at the same high bleaching and delignification is, however, observed if a second Q/PA stage is added instead of the Q/P stage (Example 7, run 4). Thus, the viscosity drop may be due to either the presence of a complexing agent specifically during the peroxy treatment or the application of a P stage after delignification with a peroxy compound. Confirmation of the latter reason can be found in Example 9 of document (3), where pulp viscosity is decreased by treatment in a Q/P-O-Q/P sequence (test 3) as against application of a Q/P-O sequence (test 2) but increased by applying a Q/P-O-Q/PA sequence (test 4).

The Board holds therefore that the conclusion to be



drawn by a skilled person from these observations is not to perform a Q/P stage after a Q/PA stage or after a first Q/P stage. Thus, if a skilled person would consider at all any application of a PA stage in combination with the Q-W-P sequence of document (2), he would be guided by the teaching of document (3) to apply it thereafter, thus giving a Q-W-P-PA sequence.

Even if one were to assume that, for whatever reason that a skilled person would, nevertheless, have considered performing a P stage after the PA stage, he would have been deterred from introducing the PA stage before any addition of a complexing agent by the teaching in document (3) that the peracetic acid might be decomposed in the presence of transition metal ions (page 7, lines 9 to 10 in combination with lines 18 to 24). Thus, there was no reason for a person skilled in the art to expect any advantage from a Q-W-P sequence after a PA stage.

- 3.4.5 The Appellant further argued that it was known from document (5) that peracetic acid itself was not catalytically decomposed in the presence of heavy metals as long as hydrogen peroxide was absent.

As appears from its title, document (5) concerns a Pulping Conference held in Boston on 1st to 5th November 1992. The document itself is dated 1992, but the actual date of publication remains unclear and could have been some time after the conference took place and hence after the earliest priority date of the patent in suit of 27 November 1992. Since the Appellant was unable to provide any evidence as to the actual date of its publication and, hence, to establish whether or not document (5) is a prior art document,

the Board would be disinclined to consider it for this reason alone.

That apart, the Board does not accept the Appellant's arguments for the following reasons:

Document (5) concerns an investigation of the effectiveness of peracetic acid as an alternative to chlorine compounds for delignification and bleaching of pulp (see title and abstract). The relevant part of document (5) on which the Appellant relied is at pages 1219, right-hand column, paragraph 3 to 1220, left-hand column, paragraph 4. There it is indicated that equilibrium peracetic acid contains high amounts of unreacted acetic acid and hydrogen peroxide which increase the cost of the peracetic acid and can damage the fibre. It is further stated that metal catalysed decomposition of hydrogen peroxide generates nonselective radical species which attack the cellulose of the pulp. It goes on to say that cellulose destruction can be combatted by removal of unreacted hydrogen peroxide by washing or by the addition of a chelating agent to sequester the metal ions which, however, is said to add to the costs of the peracetic acid. Finally, document (5) mentions a third method, namely the removal of excess acetic acid and hydrogen peroxide by distillation.

Thus, document (5) deals with the problems linked to the presence of unreacted material in the equilibrium mixture, and in particular with the problems linked to the possible generation of radicals from the unreacted hydrogen peroxide. However, the Board does not see in these paragraphs any statement saying that peracetic acid itself would not be sensitive to metal catalysed

decomposition which would in any event contradict statements to the opposite in documents (1) and (3) (document (1), page 4, paragraph 5; document (3), page 7, lines 18 to 24).

3.4.6 The Board therefore concludes that, whilst the various stages in the claimed bleaching process were in principle known from the prior art for the same purpose of effective chlorine-free bleaching and delignification, but either not in combination with peracetic acid (document (2)) or in a different sequence (documents (1) and (3)), their particular combination according to the process of Claim 1 of the patent in suit in order to improve further the quality of the product obtained by the process of document (2) was not obvious in view of the prior art documents whether considered individually or in combination.

4. *Inventive step in view of document (3) as the closest prior art*

No other result is obtained if, as alternatively suggested by the Appellant, Example 7 of document (3) is used as the closest prior art where a O-Q/PA-W-Q/P sequence (run 5) is mentioned (see 3.4.3 above). In the absence of any effect in comparison with this sequence, the problem to be solved in view of such prior art may be seen in providing another chlorine-free process for delignifying and bleaching pulp with **peracetic acid and peroxide as bleaching agents**. Whilst the problem is solved by the claimed PA-Q-W-P sequence, the Board does not see any incentive in the prior art to do so. In particular, not only document (3) itself but also document (1) both recommend treatment of the pulp during or before a PA stage with a complexing agent in

order to prevent its metal catalysed decomposition (see 3.4.2 and 3.4.3 above) and, therefore, they teach away from the idea of introducing a Q stage only after the PA stage. Nor does the skilled person find any incentive to do so in document (5) (see 3.4.4) or document (2) which does not even mention a PA stage.

5. The Board therefore holds that the process of Claim 1 is based on an inventive step as required by Article 56 EPC.

Dependent Claims 2 to 11, which refer to preferred embodiments of Claim 1, are based on the same inventive concept and derive their patentability from that of Claim 1.

## **Order**

### **For these reasons it is decided that:**

The appeal is dismissed.

The Registrar:

The Chairman:

G. Rauh

P. Krasa