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**Datasheet for the decision
of 7 February 2012**

Case Number: T 1051/09 - 3.3.03
Application Number: 98964923.1
Publication Number: 1044235
IPC: C08G 73/02, D21H 17/55,
D21H 21/20
Language of the proceedings: EN

Title of invention:

Process to reduce the AOX level of wet-strength resins by
treatment with base

Patentee:

HERCULES INCORPORATED

Opponent:

Eka Chemicals AB

Headword:

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Relevant legal provisions:

EPC Art. 83

Keyword:

"Sufficiency of disclosure - (no) - all requests"

Decisions cited:

T 0226/85, T 0435/91

Catchword:

-



Case Number: T 1051/09 - 3.3.03

D E C I S I O N
of the Technical Board of Appeal 3.3.03
of 7 February 2012

Appellant:
(Patent Proprietor)

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Decision under appeal:

**Decision of the Opposition Division of the
European Patent Office dated 2 December 2008 and
posted 12 March 2009 revoking European patent
No. 1044235 pursuant to Article 101(3)(b) EPC.**

Composition of the Board:

Chairman: B. ter Laan
Members: M. C. Gordon
C. Vallet

Summary of Facts and Submissions

I. The appeal lies against the decision of the opposition division dated 2 December 2008 and posted 12 March 2009 revoking European patent number EP-B1-1 044 235 (application No. 98 964 923.1).

Claims 1, 35, 36 and 40 of the patent read as follows:

1. A process for reducing the AOX content of a starting water-soluble wet-strength resin comprising azetidinium ions and tertiary aminohalohydrin, comprising treating said resin in aqueous solution with base to form treated resin, wherein at least 90% of the tertiary aminohalohydrin present in the starting resin is converted into epoxide. the level of azetidinium ion is substantially unchanged, and the effectiveness of the treated resin in imparting wet strength is at least as great as that of said starting wet-strength resin.
35. The water-soluble wet-strength resin obtainable by the process of any one of the preceding claims.
36. A process for preparing paper comprising:
 - (a) providing aqueous pulp slurry
 - (b) adding to the aqueous pulp slurry the water-soluble wet- strength resin of claim 35
 - (c) sheeting and drying the aqueous pulp slurry to produce paper.
40. A process for reducing the AOX content of a composition comprising halohydrin functionality comprising treating the composition with base wherein the AOX content is reduced to less than 10% of the AOX content in the untreated composition on an equal solids level basis.

II. A notice of opposition was filed on 24 January 2006 in which revocation of the patent on the grounds of Art. 100(a) EPC (lack of novelty and lack of inventive step) and Art. 100(b) (insufficiency of disclosure) was requested.

III. The decision under appeal was based on the claims of the patent as granted (main request) and three sets of claims forming first to third auxiliary requests. Claim 1 of each of the sets of claims according to the auxiliary requests was directed to a process for preparing paper.

The decision held that the patent in suit did not meet the requirements of Art. 83 EPC since it did not teach, beyond the specific examples given, how to carry out the base treatment in order to meet the requirements of the claims pertaining to conversion of tertiary aminohalohydrin present in the starting resin into epoxide (i.e. the adsorbable organic halogen (AOX) content), azetidinium content (AZE) and wet strength. On the contrary the skilled person could only establish by trial and error involving a large number of experiments whether a particular combination of parameters (temperature, type and amount of base, pH, reaction time, resin concentration) would yield the required treated resin. This constituted an undue burden. This conclusion applied to all requests under consideration.

Furthermore claim 40 of the main request was held to lack novelty (Art. 54 EPC).

Consequently the patent was revoked.

- IV. On 12 May 2009 the patent proprietor lodged an appeal against the decision, the prescribed fee being paid on the same day.

- V. The statement of grounds of appeal was received on 22 July 2009. The main request for rejection of the opposition (maintenance of the patent as granted) was maintained. Sets of claims forming four auxiliary requests were submitted.

VI. The opponent, now the respondent, replied with a letter dated 26 November 2009.

VII. On 14 October 2011 the Board issued a summons to attend oral proceedings which was accompanied by a communication setting out its preliminary, provisional opinion, in particular regarding Art. 83 EPC and Art. 54 EPC.

VIII. With a letter dated 6 January 2012 the appellant made further written submissions and filed nine amended sets of claims constituting a main request and eight auxiliary requests designated auxiliary requests 1, 2, 3, 3A, 4, 4A, 5 and 5A whereby auxiliary requests 3-5A were directed to a process for preparing paper. Claim 1 of all sets contained a feature relating to the resin treatment step, analogous to that in claim 1 of the patent as granted.

IX. Oral proceedings were held before the Board on 7 February 2012.

Following discussion, the appellant withdrew the main, first and second auxiliary requests and submitted an amended version of the third auxiliary request, i.e. the set of claims numbered 3, which thus became the main request. Claim 1 of this request read as follows:

"A process for preparing paper comprising:
(A) a resin treatment step for reducing the AOX content of a starting water-soluble wet-strength resin comprising azetidinium ions and tertiary aminohalohydrin, comprising treating said resin in aqueous solution with base to form treated resin,

wherein at least 90% of the tertiary aminohalohydrin present in the starting resin is converted into epoxide, the level of azetidinium ion is substantially unchanged, and the effectiveness of the treated resin in imparting wet strength is at least as great as that of said starting wet strength resin; and

(B) a paper-making step comprising:

- (a) providing aqueous pulp slurry
- (b) adding to the aqueous pulp slurry the treated resin produced in step (A)
- (c) sheeting and drying the aqueous pulp slurry to produce paper."

The respondent stated that it had no formal objections (Art. 84, 123(2)(3) EPC) to this request.

X. The arguments of the appellant can be summarised as follows.

(a) The key contribution of the patent to the art was the recognition that by adopting a moderate base treatment it was possible to achieve the dual effects of:

- removing AOX, i.e. organically bound halogen whilst
- retaining azetidinium ions or wet strength, which was reflected in the claim by three conditions:
 - 90% of tertiary aminohalohydrin in the starting material was to be converted to epoxide;
 - the level of azetidinium was to be substantially unchanged;

- the effectiveness of the resin at imparting wet strength was at least as great as that of the starting wet strength resin.

- (b) Considering the nature of the treatment, it was only possible to formulate the claims by way of the object to be achieved. The description and examples showed how to select appropriate treatment conditions for various resin types and explained why some examples gave better results than others. Whilst initial trials might be needed to establish the conditions for a given resin this did not constitute an undue burden. The extensive teaching, in particular the large number of examples, of the patent in suit showed that it was necessary to adopt moderate conditions. Conditions that were too mild would not result in sufficient conversion of aminohalohydrin to epoxide whilst conditions that were too harsh would fail to allow retention of azetidinium ions.

- (c) In paragraphs [0035] and [0038] of the patent in suit it was explained that various factors affected the harshness of base treatment, in particular temperature, time and pH. Whilst there were multiple permutations of parameters the examples provided sufficient teaching as to how these could be selected for various resins. The skilled person in any case knew about base treatment and was aware of how these factors influenced the outcome.

- (d) Thus from examples 25, 26 and 28 of the patent it was apparent that:

- a mild base treatment did not allow satisfactory AOX removal (example 28);
- a harsh base treatment did not allow adequate retention of AZE (example 26);
- a moderate base treatment allowed simultaneously removal of AOX and adequate retention of AZE (example 25).

Regarding specifically the requirement to retain azetidinium content, from examples 24-26 it was apparent that conditions that were mild resulted in hardly any loss of azetidinium (example 24). The moderate conditions of example 25, with a higher maximum pH resulted in more reduction of azetidinium but still retained this at an acceptable level, whereas the harsh conditions of example 26 with even higher pH resulted in an unacceptable loss of azetidinium ions.

Regarding reduction of AOX, example 18 showed that conditions that were too mild did not result in adequate reduction of AOX whereas example 17, employing moderate conditions, did provide sufficient reduction in AOX content.

The examples showed that in general conditions of 40-55°C, 5 minutes at pH 10.5-11 were suitable.

- (e) The wet-strength requirement was only to be seen as an explanatory feature. Whereas in known base treatments the wet-strength was lost, that was not the case with the present base treatment. From paragraph [0037] of the patent in suit it was

clear that AOX removal took place without loss of wet-strength.

XI. The submissions of the respondent can be summarised as follows:

(a) As followed from paragraph [0034] of the patent in suit a great many factors affected the outcome making it necessary to carry out a large number of experiments to identify appropriate conditions. For example the amount and type of polymeric aminochlorohydrin, the nature of the epichlorohydrin byproducts, the level of stabilisation acid in the resin, conditions of activation of the resin all played a role. The examples of the patent were inconsistent as some met the requirements set out in the claims, whereas others did not.

(b) The claims were not limited to any particular resins meaning that in order to meet the requirements of Art. 83 EPC the patent had to put the skilled person in the position to achieve the necessary results with any water-soluble wet-strength resin. This was however not the case.

XII. The appellant requested that the decision under appeal be set aside and that a patent be maintained on the basis of auxiliary request 3 as filed at the oral proceedings or on the basis of auxiliary requests 3A, 4, 4A, 5, 5A all submitted with the letter dated 6 January 2012.

XIII. The respondent requested that the appeal be dismissed.

Reasons for the Decision

1. The appeal is admissible.

Main request (designated "auxiliary request 3")

The main request is the set of claims filed at the oral proceedings before the Board as auxiliary request 3. Since all higher ranking requests were withdrawn, this is the main request.

Article 83 EPC

2. The claimed process is defined in a functional manner, i.e. by its outcome. Since the claimed subject-matter is not limited to particular water-soluble wet-strength resins, the question to be answered with respect to Art. 83 EPC is whether the way to achieve that outcome for any possible water-soluble wet-strength resin is disclosed in the patent in suit in such a manner that it is fit for generalisation beyond the specific examples disclosed.
 - 2.1 The only general information relating to the nature of the resin is provided in paragraph [0034] of the patent in suit which states that the amount of base varies widely from resin to resin and teaches that this is dependent on :
 - resin type
 - amount and type of polymeric aminochlorohydrin
 - the amount of epi by-products
 - the amount of stabilisation acid in the resin

- the conditions used to activate the resin.

There are however no further explanations, analyses or discussions of any of these factors, their interrelationship and how they affect the outcome. Thus all that this paragraph provides is a disclosure which while comprehensive in scope is almost devoid of any relevant information. In particular this paragraph does not amount to the provision of a teaching of a concept fit for generalisation since no consideration is presented of how the identified factors might, even in a very general manner, influence the outcome or interact with the treatment system.

According to paragraph [0035], the treatment temperature ranges from 0° to 100°C, the time from 1 minute to 24 hours and the resin solids content for base treatment from 1% up to 40%. These are very broad ranges and again there is no discussion or appreciation of their interdependence or relationship to other resin properties.

Paragraph [0038] provides the general statement that treatment conditions can be optimized for each resin to a given set of conditions, but that other conditions also give good results and that if shorter reaction time is desired then higher temperatures are needed.

Thus the sum total of the general teaching is that it is necessary to select conditions that are neither "too mild" nor "too harsh", but are "moderate" or "just right" and that the conditions have to be adapted to the resin and a list of factors which influence the reaction, in line with the argumentation of the

appellant.

2.2 As submitted by the appellant, and not disputed by the respondent, the patent in suit does indeed contain a great many examples. It is also the case that a number of different resins are employed in these examples. However the outcome of these is variable - some provide the required outcome in terms of reduction of AOX content (and also conversion of aminohalohydrin to epoxide) and retention of azetidinium groups, whereas others do not.

2.2.1 The appellant drew attention in particular to the family of examples 25, 26 and 28, which would reveal that the required "moderate" conditions to obtain simultaneously the required reduction of AOX and retention of azetidinium were provided by example 25, in which Kymene[®] 736 resin was treated:

- at 55°C for 5 minutes
- with 4.3 mmole NaOH/g resin and peak pH of 10.8 resulting in a percentage AZE of 38, a percentage aminochlorohydrin (ACH) of <2 and 165 ppm AOX.

The conditions of example 26 were however "too harsh", leading to a too great reduction in azetidinium:

- carrying out reaction under the same conditions of temperature and time, i.e. 55°C for 5 minutes
- but with 5.1 mmol NaOH/g resin and a maximum pH of 11.1

resulting in a percentage AZE of 32, a percentage ACH of <2 and an undetectable amount of AOX.

Example 28, according to the appellant, demonstrated conditions that were "too mild", i.e. did not lead to the required reduction of AOX:

- 25°C for 5 minutes
- same base concentration as example 25, i.e. 4.3 mmole NaOH/g resin but higher maximum pH namely 11.8 resulting in a percentage AZE of 37, a percentage ACH of <2 and 770 ppm of AOX.

As the respective values of the starting resin were 42%, 30% and 4820 ppm the reduction was (rounded):

Example 25: 10% AZE, >93% ACH, 96% AOX

Example 26: 34% AZE, >93% ACH, 100% AOX

Example 28: 12% AZE, >93% ACH, 84% AOX.

Apparently, from the appellant's submissions, a reduction of 10% or 12% of AZE is still considered as being "substantially unchanged".

2.2.2 Further, the appellant drew attention to examples 24 to 26 in which the effect of the "harshness" of treatment on AZE retention was demonstrated. In example 24, treatment was carried out at

- 55°C for 5 minutes
- 3.9 mmole NaOH/g resin, peak pH 10.3

which resulted in hardly any loss of AZE with a percentage of 41, a percentage ACH of <2 and an undetectable amount of AOX. Hence, the reduction was:

Example 24: 2% AZE, >93% ACH, 100% AOX.

2.2.3 The effect of the nature of the treatment on reduction in AOX content was stated by the appellant to be demonstrated by examples 17 and 18, in which however

Kymene[®] 557LX resin, hence a different resin from that of examples 24-26 and 28, was employed:

Example 17:

- temperature/time conditions of 55°C/5 minutes
 - a base concentration of 2.4 mmole NaOH/g resin
 - the reaction reaching a peak pH of 10.9
- resulting in a percentage AZE of 47.5 and a percentage ACH of 0.0 and 200 ppm of AOX.

In example 18 the same temperature/time conditions were employed but with 2.0 mmole NaOH/g resin, and a peak pH of 10.4, leading to a percentage AZE of 51.0, a percentage ACH of 1.5 and 663 ppm of AOX.

The values of the starting material being 51.6%, 8.8% and 3200 ppm, the reduction can be calculated:

Example 17: 8% AZE, 100% ACH and 94% AOX

Example 18: 1% AZE, 83% ACH and 79% AOX.

The conditions used in these examples differ significantly from those of examples 24-28, also in the concentration of base. Therefore, examples 17 and 18 cannot be compared or combined with examples 24-28 in order to obtain general information about how to achieve the result now being claimed.

2.3 Furthermore, in the arguments presented with respect to examples 17 and 18 the appellant focused solely on the AOX content, and omitted to explain what information these examples provide about other properties of the resin.

Accordingly, and apart from the question of the interpretation of "substantially unchanged" for the

level of AZE, not only has the appellant discussed unrelated examples in combination, it has furthermore restricted consideration of these examples to certain aspects in isolation. This approach is however inconsistent with the requirements of the claimed subject-matter: to obtain a set of properties in combination.

- 2.4 Other evidence, not invoked by the appellant, undermines the conclusions presented by the appellant regarding appropriate conditions based on example 25, showing these not to be generally applicable. Thus in comparative example 5 a polyamino-polyamide-epi resin containing about 13 mole% of AZE was prepared and in the related examples 21 and 22 it was base treated. In the results and discussion of examples 21 and 22 it is stated that treatment at 55°C for 5 minutes at a base concentration of 5.0 mmole NaOH/g resin and a peak pH of 11.6, i.e. a higher NaOH concentration and peak pH than employed in example 25, leads to the desired properties of the treated resin (a percentage AZE of 11.8, a percentage ACH of 0.0 and 244 ppm of AOX). According to example 22 however, the same temperature/time conditions but with a base concentration of 4.3 mmole NaOH/g resin and a peak pH of 11.2, i.e. the same base concentration as example 25 but a higher peak pH, do not lead to a desirable product, with a percentage AZE of 12.8, a percentage ACH of 5.1 and 759 ppm of AOX.

The starting resin having values of 13,1%, 54,8% and 9600 ppm, respectively, the reduction can be calculated:
Example 21: 10% AZE, 100% ACH and 97% AOX
Example 22: 2% AZE, 89% ACH and 92% AOX.

The evidence of these examples in relation to example 25 is that similar reaction conditions can lead to greatly differing outcomes in the case of different resins. The patent in suit however provides no explanation of the underlying factors leading to these differences.

- 2.5 For the sake of completeness, the Board also notes that the teaching of the patent in suit with respect to the requirement of claim 1 of maintenance of the effectiveness of the resin at imparting wet-strength properties is also insufficient. There is a discrepancy between the results depending on the aging of the paper. Only in the case of examples 21 and 22 is a consistent improvement shown. For many other examples the requirement that the wet-strength should be at least that imparted by the starting resin is not fulfilled.
- 2.6 The only conclusion that can be drawn from the above is that the conditions necessary to meet the varying requirements of the resin, in particular conversion of AOX and retention of AZE as well as maintenance of the effectiveness of the resin at imparting wet-strength properties, cannot be generalised but need to be adapted for each starting resin to be treated.
- 2.7 Accordingly the patent in suit provides no indication or teaching for the skilled person on how starting from a given resin it is possible in a directed and structured manner to identify an appropriate set of conditions. Nor does the patent provide any indication or guidance to assist the skilled person in evaluating the outcome of unsuccessful trials to identify which

modifications need to be made in order to progress towards conditions which give the desired result.

Each set of examples in the patent in suit relates to a specific resin. The appellant has failed to show that the information given in the patent relating to the nature of the resins and conditions employed would provide the skilled person with the insight sufficient to understand the interaction between these factors and how they influence the outcome so that it would be possible in a directed way, without undue burden, to converge on a set of conditions which for any given resin would yield the desired result.

Accordingly the technical teaching of the patent amounts to little more than a report that it has been found possible to provide optimised conditions enabling the various competing requirements to be met and some examples of special cases in which this was achieved. What is lacking is a generalisable teaching applicable within the scope of the claims, i.e. beyond the specific examples (cf. decision (cf. decisions T 435/91 (OJ EPO 1995, 188), in particular section 2.2.1 of the Reasons), and T 226/85 (OJ EPO, 1988, 336)).

2.8 The main request (set of claims designated "Third Auxiliary Request") therefore does not meet the requirements of Art 83 EPC and is refused.

3. *Auxiliary requests (claim sets 3A, 4, 4A, 5 and 5A).*

Since the respective claims 1 of all these sets of claims also contain a definition of the resin treatment step, these share the fate of the main request since

they do not meet the requirements of Art. 83 EPC for the same reasons.

Hence claim sets 3A, 4, 4A, 5 and 5A are also refused.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar

The Chairman

E. Görgmaier

B.ter Laan