Europäisches Patentamt
Beschwerdekammern

European Patent Office
Boards of Appeal

Office européen des brevets
Chambres de recours

Veröffentlichung im Amtsblatt ~~Ja~~/Nein
Publication in the Official Journal ~~Yes~~/No
Publication au Journal Officiel ~~Oui~~/Non

Aktenzeichen / Case Number / N° du recours : T 212/85

Anmeldenummer / Filing No / N° de la demande : 80 303 694.6

Veröffentlichungs-Nr. / Publication No / N° de la publication : 0 028 886

Bezeichnung der Erfindung / Title of invention / Titre de l'invention : Crosslinkable water-reducible coating compositions based on water-insoluble polymer microparticles in sterically stabilised dispersion

Klassifikation / Classification / Classement : C09D 3/80

ENTSCHEIDUNG / DECISION

vom / of / du 28 September 1987

Anmelder / Applicant / Demandeur :

Patentinhaber / Proprietor of the patent / Titulaire du brevet :
ICI PLC
DULUX AUSTRALIA LTD

Einsprechender / Opponent / Opposant : RÖHM GmbH

Stichwort / Headword / Référence :

EPÜ / EPC / CBE Articles 52(1), 54(1) and (2) and 56

Kennwort / Keyword / Mot clé : "Novelty - implied features"

Leitsatz / Headnote / Sommaire

EPA/EPO/OEB Form 3030 10.86

Europäisches Patentamt | European Patent Office | Office européen des brevets

Beschwerdekammern | Boards of Appeal | Chambres de recours

Case Number : T 212/85

DECISION
of the Technical Board of Appeal 3.3.2
of 28 September 1987

Appellant :
(Opponent)

Röhm GmbH Chemische Fabrik
Kirschenallee
Postfach 4242
D-6100 Darmstadt 1

Representative :

Respondents :
(Proprietors of the patent)

IMPERIAL CHEMICAL INDUSTRIES PLC
Imperial Chemical House Millbank
London SW1P 3JF
GB

DULUX AUSTRALIA LTD
McNaughton Road
Clayton Victoria 3168
Australia

Representative :

Beton, John Lonsdale
Imperial Chemical Industries PLC
Legal Department: Patents
PO Box 6
Welwyn Garden City
Herts AL7 1HD
GB

Decision under appeal :

Decision of Opposition Division of the European Patent Office dated 27 June 1985 **rejecting the opposition filed against European patent No.** 0 028 886 **pursuant to Article 102(2) EPC.**

Composition of the Board :

Chairman : P. Lançon
Members : G. Szabo
E. Persson

Summary of Facts and Submissions

I. European patent No. 28 886 was granted on 24 August 1983 with ten claims in response to the European patent application No. 80 303 694.6 filed on 20 October 1980 claiming priority of the earlier application of 22 October 1979. Claim 1 was worded as follows:-

"A crosslinkable water-reducible coating composition the film-forming material in which consists of:-

(A) crosslinkable, water-insoluble film-forming acrylic polymer particles of size less than 10 microns (10 μm) which are sterically stabilised in dispersion in a liquid blend of:-

(B) at least one water-soluble crosslinking agent for the film-forming polymer, with

(C) at least one water-soluble, non-volatile substance of molecular weight less than 1000 which is capable of participating in the reaction whereby the film-forming polymer is crosslinked but which does not appreciably dissolve or swell the particles of the said polymer,

the amount of the crosslinking agent (B) being up to 30% of the total weight of the constituents (A), (B) and (C) and the amount of the non-volatile reactive constituent (C) being up to 40% of the said total weight."

II. The Appellant (Opponent) filed an opposition against the European patent on 24 May 1984 requesting that it be revoked on the grounds that the subject-matter of the patent was not patentable for lack of novelty or at least of inventive step. The opposition was supported by the following documents:

(I) DE-A-2 644 120
(II) US-A-3 959 202

and by the subsequently filed documents:

(III) Colloidal Dispersions; Edited by J.W. Goodwin, The Royal Soc. of Chemistry, London, Special Publication No. 43, Napper, pages 99-128 (Review Symposium, University of Bristol, 8-10, September 1981)

(IV) Evans und Napper, Kolloid - Z. u. Z. Polymere, 251, 409 (1973)

(V) Heller and Pugh, Letters to the Editor, J. Chem. Phys. 22 (1954), 1778

(VI) Ottewill, R.H., "Nonionic Surfactants". M. Schick, Editor, Marcel Dekker, New York, 1967, pages 645, 646.

III. The Opposition Division rejected the opposition in a decision dated 27 June 1985. The reason given for the decision, in relation to the issue of novelty, was that the first component of the water-reducible coating composition claimed in Claim 1, namely component (A), was not disclosed in either of the two relevant citations as these documents did not refer to crosslinkable water-insoluble film-forming acrylic polymer particles of size less than 10 microns which were sterically stabilised in a dispersion. On the issue of inventive step, the Opposition Division found the claimed compositions to be non-obvious in view of the unexpected advantages of improved stability, drying and water resistance, and resistance to rust as indicated by comparative experiments provided by the Respondents over the prior art in (II), and which were unchallenged by the Appellant.

IV. The Appellant filed an appeal together with a Statement of Grounds on 14 August 1985, paying the fee at the same time.

V. The Appellant submitted substantially the following arguments:

(i) The citations had not been appropriately interpreted, since a novelty-destroying disclosure need not be explicit;

In particular, steric stabilisation was indicated in both citations by the presence of higher molecular weight molecules, such as a polyether or polyester polyol (cf. Example B of (I)) or a nonionic surfactant (cf. Example A of (I)) which could prevent flocculation, e.g. of a polymer latex, by binding on to the polymer particles.

(ii) According to the work of Napper (III) more than one mechanism of stabilisation, e.g. electrostatic and steric stabilisation, could operate simultaneously; and since the acrylic polymer dispersions in (I) also contained steric stabilising agents besides the neutralised electric charges, they represented a novelty destroying disclosure. The dispersed polymers referred to in (II), were stabilised non-ionically and could contain various polyethylene glycol ethers or esters as stabilising agents which, being polyether-or polyester-polyols, were steric in character, and their presence was thus similarly novelty destroying for the subject-matter claimed in the patent-in-suit.

The comparative experiments provided by the Respondent (on the basis of (II)), corresponding to the preparation of a polymer without steric stabilising agents but only with anionic emulsifiers, did not relate to the nearest prior art, and were in any case irrelevant in view of lack of novelty.

VI. The Respondents (Patentees) argued substantially as follows:

(i) No reasons were put forward by the Appellant in support of the assumption that polyether or polyester polyols provided steric stabilisation, nor any suggestions as to how the compounds could perform that function. The formal definition of the patent-in-suit set out the essential features of a suitable dispersing or stabilising agent, and yet none of the cited disclosures showed an agent having these features. While more than one mechanism of stabilisation may operate simultaneously, this did not mean that steric stabilisation necessarily operated in (I) in addition to the charge or electrostatic stabilisation mechanism expressly disclosed.

(ii) As regards (II), the vinyl emulsion polymers were produced using non-ionic emulsifiers in a procedure which was wholly conventional, and the skilled man would understand by this the use of emulsifiers which, while having a hydrophobic and a hydrophilic part, would still not be steric stabilisers as defined in the patent-in-suit.

VII. The Appellant requests that the decision of the Opposition Division be set aside and the patent be revoked in full, or in any case, as far as Claims 1, 2, 4-8 and 10 are concerned. However, the Appellant offers alternatively to withdraw the appeal if the Respondents are prepared to provide new claims limited unambiguously to steric stabilisation alone through amphipathic dispersing agents according to the definition on page 3, lines 6-15 and to the exclusion of stabilising charges in the dispersed polymer particles (or failing a basis for the latter, a binding declaration on the scope of the claims). The Respondents request that the decision of the Opposition Division be upheld and has not commented upon the Appellant's offer to withdraw the appeal.

Reasons for the Decision

1. The appeal complies with Articles 106 to 108 and Rule 64 EPC and is, therefore, admissible.

2. The patent-in-suit relates to water dilutable coating compositions containing dispersed, crosslinkable, film-forming polymer particles (A), a water-soluble cross-linking agent (B), and at least one water soluble substance fulfilling certain molecular weight conditions (C) - in specified proportions by weight.

3. The relevant state of the art, according to document (I), specifically describes a composition of the above type. Example B in combination with Example 6 comprises a component (A) which is a copolymer derived from 58.6% styrene, the rest being various acrylic monomers, subsequently neutralised with dimethylethanolamine. Component (B) is hexamethoxymethyl melamine, a cross-linking agent which is water soluble. As component (C), there is provided a water dispersible polyether polyol with a molecular weight of at least about 1500, being the reaction product of 1.0 mol saccharose with 20.5 mol propylene oxide. As regards component (A), the general

description in (I) is not restricted to copolymers predominantly formed from styrene. Nevertheless, the copolymers carry acid groups which are rendered water soluble by neutralisation. The cross-linking agent (B) is not discussed in detail. The water soluble polyols, i.e. component (C), may preferably have molecular weights in the range from 500 to 3000. The proportion of these components could be 70/20/10 (cf. Example 6). The purpose of the formulation was to avoid blisters due to water or volatile organic solvents when used for coatings.

4. The technical problem in respect of this state of the art was to reduce the potential to cause atmospheric pollution on curing by the release of amines, to effect the cure without having to break down electrostatic bonds and thus reducing the energy requirement, and to be susceptible of formulation at high film-forming solids contents without high viscosity whilst maintaining good application properties (i.e. stability, drying and water resistance and resistance to rust). The problem is solved according to the main claim by making copolymer (A) predominantly acrylic (cf. also, page 2, lines 46-47 in EP-A-28886) and sterically stabilised, with a particle size less than 10 µm and without the necessity of neutralising for dispersion, and by restricting the molecular weight of component (C) to less than 1000.

5. Results shown in the examples and by the experiments submitted on 15 September 1984 suggested that the aims implied by the technical problem were achieved by the claimed compositions. The films demonstrated a high solids content (74% by weight) and low energy curing (cf. Example 2) and good mechanical and resistance properties

for the coatings. In the absence of volatile components even in the form of ammonia for neutralisation, no release of undesirable air pollutants was expected. The characteristic qualities of the claimed matter were not challenged by the Appellant in this respect.

6. In view of the silence of document (I) as to particle sizes and the selection in the patent of predominantly acrylic co-polymers as well as the absence of the neutralisation step in the latter, the claimed subject-matter can be recognised as novel over (I). This is irrespective of whether or not (I) implies the use of a steric stabilisation.

7. In relation to (II), the only worked example disclosed in this document has been unequivocally admitted by the appellant to disclose only a charge stabilisation mechanism and not a steric stabilisation mechanism (submission of 23 March 1985; last paragraph). The only remaining disclosure in (II) relied upon by the appellant is that the document referred to various polyethylene glycol ethers or esters which can be used as emulsifiers in preparation of dispersed polymers by emulsion polymerisation (col. 14, lines 34-42). These methods are, however, explicitly stated to be conventional (col. 14, lines 52-53). Conventional emulsifying does not, however, necessarily imply stability on the basis of steric protection, particularly since such emusifiers are not expected to have a sufficiently high molecular weight to assure a steric stabilisation. In other words, in the absence of a worked example there is no specific or implied teaching of steric stabilisation. Thus the subject-matter of Claim 1 in the patent-in-suit must be seen as novel also over the disclosures of (II).

8. As to the inventive step with respect to (I), the modifications represented by the claimed subject-matter, i.e. the selection of particle size, of molecular weight or of the substantially acrylic character of the copolymers, do not appear necessarily to contribute in any clearly identifiable way to the quality of the product as compared with what would be expected on the basis of (I). What is more relevant is the effect of using a basically sterically stabilised polymer this time, and whether there was such a kind of stabilisation involved at all in the compositions according to document (I).

9. The disclosure of (I) is entirely silent about steric stabilisation of the polymer particles (A) in the liquid blend of (B) and (C). This is the crucial feature of dispute between the parties, which the Appellant asserts is implicitly disclosed by the presence, during the preparation of the polymer dispersion of Example B of (I), of a polyether polyol. He makes a similar assertion regarding the presence of the non-ionic emulsifier Triton X-100 in Example A of (I) even though this is not present during the preparation of the polymer dispersion but is only added after the neutralisation of the polymer with an amine. Now, it is this essential neutralisation of the carboxyl groups of the polymers of (I), preferably with an amine, which forms the step according to Claim 1 of (I), of bringing the polymers into dispersion (or solution). However, this is a charge neutralisation and the stabilisation in dispersion, which arises from it, is clearly a charge or electrostatic stabilisation and not a steric stabilisation. The Appellant has argued on the basis of (III) that more than one mechanism of dispersion stabilisation can operate simultaneously, an argument

accepted in principle by the Respondents. Thus, charge stabilisation and steric stabilisation could be present together. In the case of (I), however, this does not mean that any steric stabilisation must also be involved.

10. As to whether or not there is some steric stabilisation involved in (I), in addition to the clearly recognisable charge stabilisation, it is important to recognise that the term itself is defined in the present patent. According to this (cf. page 3 at lines 6-15), highly specialised dispersing agents are provided for the purpose, namely amphipathic polymers containing in the molecule two essential components having differing characteristics; one component being a polymer chain which is solvated by the liquid blend of components (B) and (C) and the other being a polymer chain which is not solvated by that blend and which in consequence anchors itself to the polymer particles; such a dispersing agent has the ability to form a barrier or sheath surrounding each particle, consisting of extended chains of a different polymer. The presence of this steric barrier is what prevents gross flocculation or aggregation of the particles. The polymer particles of the patent-in-suit are thus required to be "sterically stabilised" in dispersion, which must mean that the mechanism of dispersion stabilisation must be at least substantially or predominantly steric in character.

11. In contrast to this, the propylene oxide chain (s) of the saccharose polyether polyol of Example (B) of (I) would be generally not hydrophilic and to this extent non-solvated by an aqueous or hydrophilic medium, but there would, in this case, be no solvated polymer chain to provide the steric barrier; similar considerations apply to the epsilon caprolactone-diethylene glycol reaction product presented as alternative. Furthermore, Triton X-100 being an ethylene

oxide derivative (cf. (I) Example (A)) has a solvatable polymer chain, but no non-solvated polymer chain which could anchor to the polymer particles. Thus, in no case is a specific substance disclosed in (I), which on the face of it would be expected to act as a steric stabiliser to acrylic polymer particles in the sense specified in the patent.

12. Nevertheless, the Appellant asserts that the polyether polyol of Example B, corresponding to component (C) of the patent-in-suit, performs a steric stabilisation function by virtue of its very presence in the reaction medium. Such a component (C) is, however, also present as a further additive in the examples of the patent-in-suit and there is no suggestion that it would be capable of sterically stabilising the dispersion on its own or, indeed, that it has any dispersion stabilising function at all. Such a form of stabilisation is even less probable in the case of the presence of the Triton X-100 surfactant of Example (A) in (I) since this is only added after the polymer has already been brought into dispersion by neutralisation of its carboxyl groups.

Thus the kinds of compounds alleged by the Appellant to function as steric stabilisers for the polymers of (I), although conforming to the general requirement of being non-ionic, are nothing like the macromolecules referred to generally in the prior art as steric stabilisers, and are even more fundamentally different from the specific kinds of amphipathic polymers defined in the patent-in-suit for stabilising acrylic polymer systems.

13. In view of the above the burden was on the Appellant to show that there was a steric stabilisation achieved differently and operative to a relevant extent in the formulations according to (I). The reference to these other

classes in (III), including the above-mentioned polyether polyols, cannot imply, without supporting evidence, that these must make a significant contribution to the already dominant charge stabilisation. It is, therefore, necessary to conclude that the Appellant's allegation that the dispersions of (I) are sterically stabilised, has not been substantiated.

14. The question then becomes, would the claimed solution of the technical problem have been obvious to the skilled man at the relevant date? There is certainly no suggestion or teaching in (I) that the replacement of the charge stabilisation with a sterically stabilised system would provide any technical advantages related to the problem referred to above. The common general knowledge available to the skilled man seems to be that steric stabilisation has been exploited through the ages in the preparation of both water-based and, since Roman times, oil-based paints and inks. However, there has been no document cited in these proceedings which discloses any steric stabilisation in relation to any kind of polymer formulation according to those described in (I).

15. Documents (III) to (VI) cited by the Appellant, are of the nature of an academic review and deal with various theoretical models of steric stabilisation as a phenomenon. Even if some of the compositional stability aspects of sterically stabilised formulations, such as freeze/thaw stability and stability to pH change may have been anticipated by the skilled man (though there has been no submission to this effect) the required advantages of high solids/low viscosity formulation and of low energy and rapid curing with the maintenance of the application properties consequent upon using a sterically stabilised system in the context of the present invention could not have been predicted by the skilled person on the basis of

these documents. If the skilled person nevertheless contemplated the replacement of charge stabilisation altogether with a different kind of stabilisation, the consequences of such a radical change to the properties of the coating composition were unforeseeable. If, as the Appellant argues, the polymers of the invention may retain their carboxylic groups, these would not be neutralised at all and to this extent introduce a further new feature in the composition. Even if the charge stabilisation was only partial in (I), such basic modifications in the internal physico-chemical conditions of the system, could have had adverse effects on the quality of the coatings, in the absence of any earlier hint in the art about a similar use of steric stabilisation, an otherwise commonly known technique, even in a remotely analogous situation. The Appellant submitted no evidence to show that the suggested impressive properties of the claimed compositions were non-existent or no better than those obtained according to (I). Claim 1 is therefore based on an inventive step with regard to this document.

16. Whilst the Respondents compared the performance of the coatings according to the invention with those prepared by the methods disclosed in (II), the favourable results were not challenged as facts. The conclusion of the first instance that there was an invention in respect of (II) as the relevant art, must therefore be confirmed. The Appellant's argument that this was not the most relevant art in view of no steric stabilisation has compelled the Board to investigate the situation in respect of (I), as well, as a starting point, and the alleged partial steric stabilisation, assuming that this may be more relevant in the absence of similar evidence to the contrary. The conclusion, as stated above, is identical, i.e. the subject-matter of Claim 1 is based on an inventive step in respect of (I) as well. Since the Appellant was

unsuccessful in his attempt to establish any kind of steric stabilisation for the relevant prior art, there seems to be no ground to require the Respondents to limit the term "steric" stabilisation to the wording of the definition of the same in the specification.

17. The remaining Claims 2-10 are all directly or indirectly dependent on Claim 1 and their subject-matter is thus, by the same token, novel and based on an inventive step.

Order

For these reasons, it is decided that:

The appeal is dismissed.

The Registrar The Chairman

F.Klein P.Lançon