

Publication in the Official Journal ~~Yes~~ / No

File Number: T 429/90 - 3.3.1

Application No.: 81 301 261.4

Publication No.: 0 038 127

Title of invention: Multi-layer coating process involving use of aqueous basecoat composition containing crosslinked polymer microparticles and having a pseudoplastic or thixotropic character.

Classification: B05D 7/26

DECISION
of 14 July 1992

Proprietor of the patent: Imperial Chemical Industries PLC

Opponent: Akzo N.V.

Headword:

EPC Article 56

Keyword: "Inventive step (confirmed)"

Catchwords



Case Number : T 429/90 - 3.3.1

D E C I S I O N
of the Technical Board of Appeal 3.3.1
of 14 July 1992

Appellant :
(Opponent)

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Decision under appeal :

Decision of Opposition Division of the European
Patent Office dated 26 March 1990 rejecting the
opposition filed against European patent
No. 0 038 127 pursuant to Article 102(2) EPC.

Composition of the Board :

Chairman : K.J.A. Jahn
Members : R.W. Andrews
J.-C. Saisset

Summary of Facts and Submissions

I. European patent No. 0 038 127 in respect of European patent application No. 81 301 261.4, which was filed on 24 March 1981, was granted on 17 October 1984 (cf. Bulletin 84/42) on the basis of twenty claims. Independent Claims 1 and 13 read as follows:

"1. A process for the production of a multilayer protective and/or decorative coating upon a substrate surface, which comprises the steps of:

- (1) applying to the surface a basecoat composition comprising (a) a film-forming material, (b) a volatile liquid medium for the said material and (c) pigment particles dispersed in the said liquid medium;
- (2) forming a polymer film upon the surface from the composition applied in step (1);
- (3) applying to the basecoat film so obtained a transparent topcoat composition comprising (d) a film-forming polymer and (e) a volatile carrier liquid for the said polymer;
and
- (4) forming a second polymer film upon the basecoat film from the composition applied in step (3),

characterised in that the constituents (a) and (b) of the basecoat composition are provided by a dispersion in an aqueous medium of crosslinked polymer microparticles which have a diameter in the range 0.01 to 10 μm , are insoluble in the said aqueous medium and are stable towards gross flocculation, the dispersion having a pseudoplastic or thixotropic character.

13. A basecoat composition suitable for use in the process according to Claim 1, the composition comprising:

- (A) a film-forming material,
- (B) a volatile liquid medium for the said material, and
- (C) pigment particles dispersed in the said liquid medium;

characterised in that the constituents (A) and (B) are provided by a pseudoplastic or thixotropic dispersion in an aqueous medium of polymer microparticles which have a diameter in the range 0.01 to 10 μm , are insoluble in the said aqueous medium and are stable towards gross flocculation, the dispersion having been produced by the steps of (i) polymerisation in an aqueous medium in the presence of a stabiliser of a first mixture of monomers selected so as to give rise to particles of crosslinked polymer, (ii) polymerisation in the same medium and in the presence of the said crosslinked particles of a second mixtures of monomers selected so as to give rise to a non-crosslinked polymer which is capable of forming a water-soluble salt derivative and (iii) subsequent formation of the said salt derivative in the presence of the same aqueous medium."

II. Notices of opposition, which were filed on 17 July 1985, requested the revocation of the patent on the ground that its subject-matter did not involve an inventive step. The oppositions were supported, inter alia by the following documents:

- (1) EP-A-0 001 489
- (3) DE-B-2 557 434; equivalent to US-A-3 953 643 (3a),
and
- (4) DE-C-2 742 282; equivalent to US-A-4 062 823 (4a).

After expiry of the time allowed for filing notice of opposition, an objection with respect to lack of novelty was raised and the following document was also referred to:

(2) US-A-4 075 141.

In a communication filed on 26 September 1989, Opponent I withdrew his opposition.

- III. By a decision issued on 26 March 1990, the Opposition Division rejected the opposition. The Opposition Division held that the claimed subject-matter was novel and that the proposed solution to the problem of controlling loss of diluent from a basecoat composition due to evaporation between the spray gun and substrate was inventive in the light of the cited prior art.
- IV. An appeal was lodged against this decision on 22 May 1990 and the prescribed fee duly paid. In his statement of grounds of appeal filed on 25 July 1990 and during the oral proceedings held on 14 July 1992, the Appellant alleged that the subject-matter of Claims 1 to 12 did not involve an inventive step irrespective of whether the pre-characterising part of Claim 1, the basecoat or the aqueous basecoat/topcoat system known from document (3a) was used as the starting point for its assessment.

The Appellant argued that the skilled person faced with the deficiencies of the basecoat/topcoat system of document (3a) would be led directly by the teaching of documents (1), (2) or (4a) to modify this known basecoat by the inclusion of cross-linked microparticles in, and the provision of a pseudoelastic/thixotropic character to this known basecoat. The Appellant contended that although documents (2) and (4a) were concerned with topcoat

compositions and made no reference to humidity conditions, they were relevant since they disclosed the effects of including polymer micro-particles in paints. According to the Appellant the problems of sagging and solvent popping addressed and solved by documents (2) and (4a) are the same ones that result from the inability to selectively control the loss of diluent from waterborne basecoats. Therefore, the skilled person would include polymer microparticles in waterborne basecoats with a reasonable expectation of successfully solving the problems associated with such systems.

Although the Appellant admitted that certain compositions within the scope of claims possessed an unexpected combination of properties, he maintained that the scope of Claim 1 was too broad.

- V. The Respondent submitted that document (3a) constitutes the closest prior art since this is the only cited document directed specifically to waterborne basecoat/clearcoat systems capable of being applied over a range of humidities. In the light of this prior art, the Respondent saw the technical problem as providing a further improved basecoat/clearcoat system which can be sprayed at a wide range of relative ambient humidities while at the same time avoiding strike-back and providing in the case of metal flake pigments a proper "flip tone".

The Respondent argued that no justification exists for combining document (3a) with any of documents (1), (2) or (4a). Therefore, in the absence of such justification, it was not permissible to combine the teaching of these documents with a view to showing lack of inventive step. Furthermore, combining the teaching of documents (1) and (3a) by replacing the polymer combination of document (3a) would remove a key feature of this document.

Finally, the Respondent maintained that there was no justification for concluding that the attainment of satisfactory spray application necessarily means that the spray composition must be thioxotropic or pseudoplastic.

- VI. 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. Alternatively, as auxiliary requests, the Respondent requested that the patent be maintained on the basis of claims in accordance with his second, third, fourth or fifth auxiliary request filed on 19 March 1992.

- VII. At the conclusion of oral proceedings, the Board's decision to dismiss the appeal was announced.

Reasons for the Decision

- 1. The appeal is admissible.

- 2. The patent in suit relates to a process for the production of a multi-layer protective and/or decorative coating upon a substrate surface by first applying, preferably by spraying, a waterborne basecoat to the surface of the substrate and then applying, again preferably by spraying, an unpigmented topcoat; i.e. a so-called clearcoat. For economic reasons the waterborne basecoat must be capable of being sprayed under a wide range of relative ambient humidities.
 - 2.1 Document (3a), which is considered to represent the closest prior art, also relates to a waterborne basecoat/clearcoat system which is capable of being

applied with minimal humidity control (cf. Claim 1 in combination with column 2, lines 22 to 25). This is in contrast to the waterborne paint system introduced by one leading motor vehicle manufacturer which required careful control of humidity in the spray area to achieve successful spray application. However, the waterborne basecoat/clearcoat system disclosed in document (3a) was found to be unsatisfactory with respect to its performance at relatively high ambient humidities. Additionally, when the basecoat contained metal flake pigments a good "flip-tone" effect was not obtained even when the basecoats were sprayed at a relative humidity of 48%.

- 2.2 In the light of this closest prior art, the technical problem underlying the disputed patent is to be seen in providing an improved process for the production of multi-layer coatings. The improvement with respect to the process of document (3a) is an increase in the stability of the spraying process towards variation in relative humidity while, at the same time, avoiding strike-back and achieving a good "flip-tone" effect when the basecoat contains a metal flake pigment.

Strike-back is the phenomenon by which spray application of the clearcoat disturbs the surface of the basecoat and, particularly, in metallic flake-containing basecoats adversely affects appearance of the film. "Flip-tone" is the effect seen in metallic paints where a painted surface appears to be light when viewed perpendicularly but darker when viewed at an oblique angle.

- 2.3 According to the disputed patent, this technical problem is essentially solved by first applying to the surface a basecoat composition comprising a dispersion in an aqueous medium of cross-linked polymer microparticles which have a diameter in the range 0.01 to 10 μm , are insoluble in the

aqueous medium and are stable to gross flocculation and then applying to the basecoat film a clearcoat composition comprising a film-forming polymer and a volatile carrier liquid therefor. The said dispersion must also have a pseudoelastic or thixotropic character.

- 2.4 After comparing the painted plates submitted with the Statutory Declaration of A.J. Backhouse in connection with the opposition of Herberts GmbH filed on 29 December 1986, the Board is satisfied that the above-defined technical problem has been solved. In particular, the appearance of plates Nos. AJB/1, AJB/2, AJB/3 and AJB/4 (sprayed at relative humidities of 48% or between 45% and 50%) and AJB/17 (sprayed at a relative humidity of between 60% and 65%), which were coated with basecoats and clearcoat compositions in accordance with the disclosure of document (3a), was unsatisfactory especially as regards the "flip-tone" effect and sagging. In contrast, the appearance of plates Nos. AJB/15 and AJB/16 (sprayed at relative humidities of 50% and between 45% and 50%), and AJB/22 and AJB/23 (sprayed at relative humidities of 70% and between 60% and 65%), which were coated with basecoats and clearcoats in accordance with the disputed patent, was completely satisfactory.
3. The only issue to be decided in this appeal is whether the proposed solution to the technical problem underlying the disputed patent is inventive.
- 3.1 As previously mentioned document (3a) discloses a multi-layer coating process involving the use of waterborne basecoats. Accordingly, this document describes a method of coating a substrate comprising first applying to it a coating composition comprising a dispersion in an aqueous solution of a water-soluble amine of a mixture of from 6 to 60 parts by weight of particulate pigments and from 40

to 94 parts by weight of a film-forming thermosetting paint binder, at least partially drying the first coating, applying to the coated substrate a second coating composition comprising an aqueous dispersion of a film-forming thermosetting resin capable of giving a substantially transparent coating upon baking and baking the cured surface. The paint binder of the base coat consists of 100 parts by weight of acrylic paint binder resins containing about 5 to about 95 parts by weight of a solution polymer which is a carboxy-functional acrylic copolymer that is at least partially neutralised with the aqueous solution of the water-soluble amine, is soluble in the aqueous medium, has an average molecular weight of in the range of about 3,000 to about 20,000 and has a Tg in the range of -15°C to 50°C; about 5 to about 95 parts by weight of an emulsion polymer which is an acrylic copolymer having functionality selected from carboxy functionality and hydroxy functionality that is essentially insoluble in the aqueous solution, has an average molecular weight in the range of about 3,000 to about 20,000 and has a Tg in the range -15°C to 50°C; and about 15 to 35 parts by weight of an amino cross-linking agent for the solution and emulsion polymers (cf. Claim 1). Therefore, the basecoat of this prior art method is based on a blend of low molecular weight solution polymer and a low molecular weight emulsion polymer, neither of which is cross-linked. Furthermore, this document is wholly silent with respect to the rheological character of the basecoat.

In the Board's judgment, the skilled person faced with the problem of improving this prior art process would not initially contemplate changing the essential novel component of the basecoat of document (3a), viz. the mixture of a low molecular weight emulsion polymer and a

low molecular weight solution polymer (cf. column 5, lines 20 to 35), unless there was some incentive to do so.

- 3.2 Document (1) discloses a process for the preparation of a sterically stabilised aqueous dispersion of cross-linked polymer particles (cf. Claim 1 in combination with the Examples). This document suggests that the rheological properties, such as a high degree of thixotrophy, of the dispersion obtained by the disclosed process makes them useful, for example, in the control of aqueous paints to be applied by spraying or by brush (cf. page 11, lines 31 to 34). However, the document is totally silent with respect to waterborne basecoats and provides no incentive to combine its teaching with that of document (3a) or any indication that the use of these cross-linked polymer microparticles in waterborne basecoats would solve the present problem.

The Appellant sees an incentive to combine the teaching of documents (3a) and (1) in the disclosure in the latter document of the thixotropic character of the aqueous dispersion of the polymer microparticles disclosed therein. However, according to the uncontested statement of A.J. Backhouse in paragraph 9 of his Statutory Declaration filed on 29 December 1986 in connection with the opposition by Herbert's GmbH, one leading metallic basecoat used by a major motor vehicle manufacturer is a newtonian liquid. Furthermore, the basecoats of Experiment 1 was pseudoplastic and that of Experiment 4 was almost a newtonian liquid, but in both cases the spray application was poor. On the other hand, the basecoat of Experiment 5 was a newtonian liquid and sprayed well (cf. Plate Nos. AJB/1, AJB/4 and AJB/5; submitted with the above-mentioned Declaration).

Therefore, it cannot be justified to conclude that the attainment of satisfactory application merely requires that the spray composition must be pseudoplastic or thixotropic. Moreover, there is even less justification for concluding that the provision of this rheological character to the basecoat composition would give compositions which could be satisfactorily sprayed at relative ambient humidities in the order of 65% to 70% (cf. plate Nos. AJB/5, AJB/18, AJB/12 and AJB/20).

3.3 Document (2) concerns coating compositions comprising a carboxylic acid amide interpolymer and cross-linked polymeric microparticles (cf. column 1, lines 63 to 67). This document relates solely to topcoats and is concerned with providing adequate film thickness with two instead of the normal three coatings (cf. column 1, lines 27 to 46). The resulting films are also resistant to solvent popping and sagging (cf. column 1, lines 47 to 54). According to column 6, lines 32 to 42, the liquid medium containing the amide interpolymer and cross-linked polymeric microparticles may be either organic solvent based or aqueous based or may consist of mixtures of water-miscible solvents and water.

In order to minimise or eliminate sagging and solvent popping when utilising waterborne compositions, it was necessary to employ certain additives, such non-volatile, water soluble or water-dispersible polyether polyols or polyester polyols having a molecular weight of at least 300 (cf. column 6, lines 43 to 65). Thus, in aqueous systems the cross-linked polymeric microparticles are by themselves not capable of preventing sagging. It is also stated that these polyols function as co-solvents and lower the viscosity of the composition (cf. column 8, lines 17 to 21). This would clearly not lead the skilled

person towards basecoats having pseudoelastic or thixotropic character.

In view of the difference between topcoat systems and basecoat/clearcoat systems and the fact that document (2) is only concerned with compositions based on carboxylic acid amide interpolymers, there is no reason why the skilled person should extract only the disclosure relating to cross-linked polymeric microparticles from this document and combine it with that of document (3a).

Therefore, in the Board's judgment the disclosure of document (2) would not be of any assistance to the skilled person faced with the problem of improving the method of producing a multi-layer coating of document (3a).

- 3.4 Document (4a) discloses an aqueous paint dispersion comprising an aqueous solution of a water-soluble amine containing as film-forming components (1) about 30 to 70 parts by weight of a solution polymer which is a carboxy-functional copolymer of acrylic monomers that is at least partially neutralised with a water-soluble amine, is essentially soluble in said aqueous solution, has a number average molecular weight of from about 3,000 to 20,000 and has a glass transition temperature of from -15°C to 50°C and (2) about 70 to 30 parts by weight of an emulsion polymer which is a carboxy-functional, hydroxy-functional or carboxy-and hydroxy-functional copolymer of acrylic monomers that is derived from about 98 to about 99.5 mole percent of monoethylenically unsaturated monomers consisting of acrylates, methacrylates, acrylic acid, methacrylic acid and vinyl hydrocarbons and about 0.5 to about 2 mole percent of multiolefinically unsaturated monomers consisting of diacrylates, dimethacrylates, triacrylates, trimethacrylates, tetracrylates, tetramethacrylates and divinyl hydrocarbons and which

is essentially insoluble in said aqueous solution, has a glass transition temperature of in the range -15°C to 50°C and has a bimodal molecular weight distribution and comprises (a) a noncross-linked fraction having an average molecular weight in the range of about 3,000 to about 20,000 and (b) a cross-linked fraction constituting a gel and (3) an amino cross-linking agent which is a urea-formaldehyde resin or melamine formaldehyde resin in an amount in the range of about 15 to about 35 weight percent of the sum of the weights of the solution and emulsion polymers (cf. Claim 1 in combination with column 4, lines 43 to 47). From the examples it is clear that this document is concerned with topcoats not basecoat/clearcoat systems. According to column 1, lines 44 to 55, the purpose of including the cross-linked polymeric microparticles is to improve the resistance to crater formation and dirt pick-up of water-based paints comprising a mixture of solution and emulsion polymers and an amine. Thus, this document would not provide the skilled person with any teaching relating to the present technical problem of improving the tolerance of waterborne basecoats to variations in humidity, of avoiding strike-back or of controlling the lay-down of the metal flake to obtain good "flip".

There is no disclosure in this document to indicate that the function of the cross-linked polymeric microparticles is to control pseudoelasticity or thixotropy. On the contrary, the references to the measurement of viscosity of the enamels using a Ford Cup No. 4 (cf. Examples 1, 12 and 14) indicates that they are not pseudoelastic or thixotropic.

The disclosure of this document taken by itself would not provide the skilled person with the slightest indication that the solution to the technical problem of improving

the process of document (3a) lies in founding the basecoat formulation on a dispersion of cross-linked polymer microparticles having a diameter in the range of 0.01 to 10 μm , which dispersion has pseudoelastic or thixotropic character.

In view of the fact that documents (3a) and (4a) are concerned with different technical problems and relate to different types of paints, there is, in the Board's judgment, no justification or incentive for combining the teaching of these two documents.

4. Therefore, for the above reasons, the proposed solution to the above-defined technical problem is inventive. Hence, Claim 1 and dependent Claims 2 to 12, which relate to preferred embodiments of the process of Claim 1, are allowable.

5. The Appellant, on whom the burden of proof rests, has not provided any evidence which would lead the Board to doubt that the above-defined technical problem is not successfully solved throughout the claimed area (cf. Decision T 219/83, OJ EPO 1986, 211, particularly paragraph 12). Since the Appellant has failed to support his allegation, the Board sees no reason to restrict the scope of the present Claim 1).

6. Claim 13 relates to a waterborne basecoat composition suitable for use in the process according to Claim 1 comprising pigment particles and a pseudoplastic or thixotropic dispersion in an aqueous medium of polymer microparticles which have a diameter in the range 0.01 to 10 μm , are insoluble in the aqueous medium, are stable to gross flocculation and which has been produced by a specified process.

- 6.1 In view of the above-mentioned evidence, the Board is satisfied that compositions in accordance with this claim successfully solve the technical problem of improving the basecoat compositions of document (3a) with respect to their tolerance to variations in humidity. In the multilayer coating system, these basecoats are responsible for avoiding strike-back and, in the case of basecoats containing metallic pigments, for achieving a good "flip-tone" effect.
- 6.2 For the reasons given above for the process according to Claim 1, the Board finds that the subject-matter of this Claim 13 involves an inventive step. Dependent Claims 14 to 20, which relate to preferred embodiments of the composition of Claim 13, are also allowable.
6. In view of the above findings it is neither necessary to consider the Respondent's evidence with respect to the commercial success of basecoat/clearcoat systems of the disputed patent nor his second, third, fourth and fifth auxiliary requests.

Order

For these reasons, it is decided that:

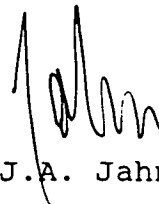
The appeal is dismissed.

The Registrar:



P. Martorana

The Chairman:



K.J.A. Jahn