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D E C I S I O N
of 26 July 1999

Case Number: T 0601/96 - 3.3.3

Application Number: 90112156.6

Publication Number: 0406671

IPC: C08J 11/24

Language of the proceedings: EN

Title of invention:
Water-soluble or water-dispersible polyester sizing compositions

Applicant:
The Seydel Companies, Inc.

Opponent:
-

Headword:
-

Relevant legal provisions:
EPC Art. 54, 56, 84

Keyword:
"Claims - clarity (yes) - product-by-process"
"Novelty - combination of product and process features (yes)"
"Inventive step - remote technical field"

Decisions cited:
-

Catchword:

-

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Case Number: T 0601/96 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 26 July 1999

Appellant: The Seydel Companies, Inc.
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Representative: Neubauer, Hans-Jürgen, Dipl.-Phys.
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Decision under appeal: **Decision of the Examining Division of the European Patent Office posted 30 January 1996 refusing European patent application No. 90 113 156.6 pursuant to Article 97(1) EPC.**

Composition of the Board:

Chairman: C. Gérardin
Members: A. Däweritz
S. Stephens-Ofner

Summary of Facts and Submissions

I. European patent application No. 90 112 156.6, filed on 26 June 1990, claiming the priority of 27 June 1989 of an earlier application in the United States of America (372015) and published under No. 0 406 671 on 9 January 1991, was refused by a decision issued in writing on 30 January 1996.

The decision was based on a set of 6 claims submitted with a letter dated 13 April 1995.

Claims 1 and 5 read as follows:

"1. A water soluble or water dispersible polyester resin, comprising the reaction product of 20-50% by weight of waste terephthalate polymer, 10-50% of at least one glycol, optionally 5-25% of at least one oxyalkylated polyol and 20-50% of a polycarboxylic acid having at least two carboxyl groups or anhydride thereof."

"5. The composition of claim 1, which is further neutralized with a base selected from the group consisting of potassium hydroxide and ammonium hydroxide."

Claims 2 to 4 and 6 related to preferred embodiments of the resin according to Claim 1, whereby Claim 6 however contained a reference to "(t)he composition of claim 1".

II. The reason for the decision was lack of clarity as well as lack of novelty and inventive step with regard to

the disclosure of the following documents:

D1: EP-A-0 152 915,

D2: BE-A-0 730 200, and

D3: GB-A-1 146 641.

Although objections under Article 123(2) EPC were raised by the Examining Division, these were explicitly stated not to form the basis for their decision.

The reasons for the decision can be summarised as follows:

(i) The expression "water soluble or water dispersible" was unclear, because the extent (i.e. the concentration) to which the resin should be soluble or dispersible was not defined in the claims. Moreover, in some of the worked examples coarse powders were obtained, whereas others related to solutions in alkali rather than in water. In fact, it was supposed that at least some of the carboxyl groups in the polyester resins had to be converted to carboxylate groups to render the resin soluble in an aqueous medium.

(ii) It could not be determined whether the final degradation product was prepared from "waste" terephthalate polymer or whether it was prepared directly from the respective acids and polyols. This was supported by the fact that "virgin terephthalate resins" were also contemplated as

suitable starting materials.

(iii) Claims 1 to 3 lacked novelty because they encompassed a reaction product of 50% polyethylene terephthalate (**PET**), 13.5% ethylene glycol (**EG**) and 36.5% isophthalic acid (**IA**) which could not be distinguished from a polyester prepared by direct esterification of equimolar amounts of terephthalic and isophthalic acids with ethylene glycol. Such polymers were undoubtedly known e.g. from D2. Neither the water solubility or dispersibility, nor the characterisation "waste" could serve as a distinguishing feature to the state of the art.

(iv) Assuming novelty to be achieved, D1 should be considered to represent the closest state of the art. It related to degradation products of PET obtained by digesting scrap polymer with a combination of polyol and polycarboxylic acid. Preferred polyols and acids included diethylene glycol (**DEG**) and **EG** as well as **IA** and trimellitic acid (**TMA**). The acid number of the products covered the range contemplated by the Applicant. Although the amount of digesting acid in the worked examples of D1 were lower than the amounts required by Claim 1, the molar ranges of the components used in D1, when recalculated as weight ranges, overlapped with those as defined in the claim.

(v) The subject-matter of the claims, if limited and clarified, would only represent an arbitrary

selection within the general teaching of D1. Such a selection would not involve an inventive step in the absence of any evidence for the criticality of the particular ranges and starting compounds mentioned in the claims and for an unexpected technical effect. Alkoxylated triols were among the preferred digesting polyols in D1, and D3 taught alcoholysis of PET with a combination of diol and triol. Further, a product having an acid number as high as 20, as addressed in D1, would necessarily have the same kind of solubility as the claimed resins.

III. On 3 April 1996, a Notice of Appeal was lodged by the Appellant (Applicant) together with payment of the prescribed fee. The Statement of Grounds of Appeal was received on 3 June 1996 in which the Appellant disputed the reasons for the refusal and indicated its preference for appeal proceedings in written procedure only.

Together with the Statement of Grounds of Appeal a new set of claims was submitted.

(i) Each of the independent claims referred as starting compounds to 20 to 50% by weight of waste terephthalate polymer, 10 to 40% of at least one glycol and, optionally, 5 to 25 % by weight of at least one oxyalkylated polyol.

(ii) Claims 1 and 3 further required the presence of 3 to 65% of a polycarboxylic acid having at least two carbonyl groups or derivative thereof and mixtures thereof with the proviso that such

polycarboxylic acid or derivative cannot be terephthalic acid.

(iii) Claims 2 and 4 further required the presence of 3 to 65% of a polycarboxylic acid having at least two carbonyl groups or derivative thereof being selected from isophthalic acid, trimellitic acid or trimellitic anhydride and mixtures thereof.

(iv) Additionally, Claims 3 and 4 were limited to resins having a minimum acid value of 20.

IV. However, in view of the number and the complexity of the objections caused by the wording of these claims, the Board decided to summon the Appellant to oral proceedings on 20 July 1999 and issued a detailed communication pursuant to Article 11(2) of the rules of procedure of the Boards of Appeal.

V. Following several rewordings of the claims and personal consultations with the Rapporteur, the Appellant filed a new Claim 1 on 8 July 1999 and new Claims 2 to 6 on 22 June 1999, on which the present decision is based. These claims read as follows:

"1. A water soluble or water dispersible polyester resin obtainable by

reacting

a) 20 - 50 % by weight of virgin and/or waste terephthalate polymer,

b) 10 - 40 % by weight of at least one glycol,

c) 5 - 25 % by weight of at least one oxyalkylated polyol, and

d) 20 - 50 % by weight of isophthalic acid

in such a way that said components a), b) and c) are heated above about 150°C, and that the thus-obtained intermediate product is further heated with said component d) at a temperature of at least 150°C.

2. A water soluble or water dispersible polyester resin according to Claim 1, characterized in that said polyester resin is further reacted with 3 to 15% by weight of trimellitic acid or trimellitic anhydride.

3. A water soluble or water dispersible polyester resin according to Claim 1 or 2, characterized in that the reaction was carried out in the presence of monobutyl stannoic acid or a tetra alkyl titanate, or a mixture thereof.

4. An aqueous solution or dispersion of a water soluble or water dispersible polyester resin according to one of the Claims 1 to 3, characterized in that said polyester resin is dissolved or dispersed in water with an alkali metal or ammonium hydroxide or carbonate.

5. An aqueous solution or dispersion of a water soluble or water dispersible polyester resin according to Claims 4, characterized in that said polyester resin is diluted to a resin solids content of 0.5 to 5%.

6. Fiber, fabric or paper coated with the water-soluble or water-dispersible resin according to one of the Claims 1 to 3."

VI. In support of the allowability of these claims the

Appellant argued essentially as follows:

(i) D1 representing the closest state of the art also referred to the recycling of terephthalate polymers by using glycol, polyol and isophthalic acid. The acid constituents were however present in lower amounts. Hence, the citation described a polyester polyol which was hydroxyl terminated and useful for making foams at which D1 aimed. However, due to the lack of carboxyl function the known polymer was not soluble or dispersible in water, in contrast to the carboxyl terminated polyesters according to the invention.

(ii) Organic solvents were necessary if solutions or dispersions of the known polymer were required for certain applications. This caused environmental problems and higher costs.

(iii) Starting from terephthalate polymer which was not biodegradable, the problem to be overcome was therefore to make available a reaction product which did not cause environmental problems and was cheap.

(iv) The solution found was not obvious for a skilled person with regard to D1, because the use of particular amounts of terephthalate resin, glycol, polyol and isophthalic acid resulted in polymers of different structure which were water soluble or dispersible, contrary to the known products. The carboxyl functional products according to the invention were not useful for the preparation of foams. Moreover, the products

included definite amounts of oxyalkylated polyols which additionally imparted hydrophilic character and less crystallinity, so that the polymers were less likely to precipitate or plate out from solution.

(v) D1 did not provide any incentive to solve the above problem but would lead the skilled person away from the preparation of water soluble or dispersible polyesters.

(vi) D2 did not disclose a polyester resin having a defined amount of terephthalate polymer, polyol, glycol and isophthalic acid, nor did it give any hint to prepare water soluble or dispersible polyester resins necessary for sizing material.

(vii) D3 showed a method for manufacturing a polyester resin but did not include the use of isophthalic acid. The plastic powders were to have improved thermal and chemical resistance. Solubility or dispersibility in water were not considered at all.

(viii) The feature of solubility and dispersibility in water was based on the presence of both carboxylated and oxyalkylated moieties. Lack of oxyalkylated moieties derived from oxyalkylated polyols led to no dispersibility, poor dispersions or milky dispersions. The carboxylated moieties were derived from isophthalic acid and, optionally, trimellitic acid by reaction with free hydroxyl groups.

VII. Since, for the reasons set out below, the present claims not only overcome the previous objections, but also satisfy all other relevant requirements of the EPC, there was no need to hold the scheduled oral proceedings.

VIII. The Appellant requested that the decision under appeal be set aside and that a patent be granted based on Claim 1 as submitted on 8 July 1999 and Claims 2 to 6 as submitted on 22 June 1999, respectively.

Reasons for the Decision

1. The appeal is admissible.

2. The requirements of Article 123(2) EPC are met by the new claims. Claim 1, which is now drafted as a product obtainable by a two-step process, is based on the combination of Claims 1 to 3, as originally filed, in conjunction with page 3, lines 5 to 7 and 31 to 34. Claims 2 to 5 are based on original Claims 4, 12, 14 and 16, respectively. Claim 6 is based on original Claim 35 and page 2, lines 19 and 20 in conjunction with lines 7 to 11.

3. *Clarity*

3.1 According to page 1, first paragraph, page 5, lines 11 to 27 and Claim 6, the application relates to the field of sizing fibres, fabrics or paper. The polymers as claimed are referred to as being "water soluble or water dispersible". The Examining Division took the view that this expression was unclear.

3.2 In "Kleines Wörterbuch der Anwendungstechnik", Hoechst AG, Frankfurt, 1975, pages 109 and 164, the keywords "Leimen", "Schlichten (Leimen)" and "Schlichtemittel", corresponding to the English and French terms "to size, sizing, sizing agent" and "encoller, encollage, produit d'encollage", respectively, refer *inter alia* to "verseifte Harze, ®Tylose usw." (saponified resins, ®Tylose etc.) and to "wasserlösliche Cellulosederivate (z.B. ®Tylose)" (water soluble cellulose derivatives (e.g. ®Tylose)), "wasserlösliche Kunststoffe (...) und Kunststoffdispersionen" (water soluble polymers and polymer dispersions) as materials used in these treatments.

3.3 In Saechtling, International Plastics Handbook, 2nd edition, Hanser, München, 1987, page 156, Chapter "4.1.1.11. *Water-soluble polymers*", the following explanation is given: "General applications of such solutions: ... textile sizes and finishes, paper sizes, ... (3) Polyacrylic acid salts and similar copolymers ... carboxymethyl cellulose (CMC ...) ... CMC solutions are compatible with alkalis and alkali salts and are not precipitated on warming, as they are on the addition of acids and solvents ..."

3.4 Moreover, examples in the application, which were obviously carried out in accordance with the invention, describe resins which are dissolved in aqueous ammonia to a solids content of 25 % (Example 1), in aqueous potassium hydroxide to a solids content of 20% (Example 3) or form a stable dispersion with dilute sodium hydroxide solution (Example 2) and can be diluted with water to solids contents of 1 to 3 % by weight and used as sizing agents for various fibres and

fabrics (Example 4). Moreover, on page 5, lines 8 to 10 and 34 and 35, values of solids contents are given to which the solutions or dispersions can be adjusted.

3.5 In the decision under appeal (cf. point 5c)) it is assumed that at least some of the carboxyl groups in the polyester resin must be converted to carboxylate groups by means of an appropriate base to render the resin soluble in an aqueous medium.

3.6 According to the Brönsted definition of acids and bases, water is a base in comparison to more acidic compounds such as carboxylic acids. Hence, a sharp line between water and aqueous solutions having a pH-value other than 7 can hardly be drawn. In each case a protolysis reaction will take place.

3.7 It follows that a person skilled in the art would have been aware of the fact that carboxylic groups-containing polymers are sensitive to pH and, whilst being soluble under basic conditions, they may be less so under neutral or acidic conditions. Moreover, the polymer according to the present claims contains further components that can also interact with the aqueous medium, i.e. oxyalkylated polyol moieties.

3.8 Taking into account the specific process conditions required in Claim 1, the Board is therefore satisfied that the feature "water soluble or water dispersible polyester resin" is sufficiently clear for a person skilled in the art and that, consequently, the combination of product features and process features represents an adequate characterisation of the resin. For these reasons the Board has come to the conclusion

that the requirements of Article 84 EPC are met.

4. *Novelty*

4.1 Claim 1 concerns a water soluble or water dispersible polyester resin defined in terms of the percentages of the components used and the process according to which it can be prepared. The process requires four essential components, (a) terephthalate polymer, (b) at least one glycol, (c) at least one oxyalkylated polyol and (d) isophthalic acid. Component (d) is reacted with the intermediate product previously obtained in the first step by reacting components (a), (b) and (c) with each other. It is common general knowledge that the structure of the resin can be influenced by both the amounts of the constituents and the sequence and the time of their addition to the reaction mixture. Hence, the argument that the resulting polyester resin is terminated by carboxylate rather than by hydroxyl groups appears acceptable, because it is consistent with the solubility properties discussed above, which are considered as a functional feature to be met by the claimed resin.

4.2 According to its page 1, lines 4 to 8, D1 relates to polyester polyols which are the hydroxyl-terminated digestion products of (A) polyalkylene terephthalate polymers and (B) polycarboxylic acid-containing polyols. The polyols are useful in the preparation of cellular foam materials, in particular for polyisocyanurate and polyurethane foams. These foams can be prepared by mixing together organic polyisocyanates with the above polyol mixtures (and optionally further polyol), catalyst and blowing agent

(page 11, lines 6 to 23).

4.2.1 According to Claim 1, component (B) is derived from a digesting polycarboxylic acid component (B-1) and a digesting polyol component (B-2). (B-1) is required to have ring units with two -COO- groups on adjacent or alternate ring positions. A preferred digesting polyol contains o-phthalic, isophthalic and/or trimellitic acid residues (Claim 1, page 4, line 16 to page 5, line 6).

Various types of (cyclo)aliphatic, aromatic, araliphatic and/or heterocyclic polyols (B-2) are mentioned, including glycols, polyoxyalkylene glycols and alkoxylated triols (page 6, line 10 *et seq.*). Aliphatic dihydric alcohols are preferred. Examples for such compounds are alkylene diols and glycol ethers. Mixtures of two or more polyols may be used. Preferred polyols are polyoxyethylene glycols, diethylene glycol and dipropylene glycol, with the latter two glycols being especially suitable (page 7, lines 1 to 3).

4.2.2 All ingredients for the digesting step can be charged at the same time to the reaction vessel and thereupon reacted together. Alternatively, the polyalkylene terephthalate is first reacted with a polyol and the polycarboxylic acid is subsequently added to the reactor and the reaction continued to completion. Various mixtures of digesting reactants, such as mixtures of diols, and polyester polyols can be introduced together in the reactor (page 6, lines 4 to 15). The reaction is deemed to be complete when the sample being digested is dissolved (page 9, line 36 to page 10, line 8).

4.2.3 The products are further characterised on page 10, lines 13 to 35: Apart from their Brookfield viscosities (500 to 50000 cP (mPas) at 25°C), hydroxyl number values of about 700 to about 120, preferably 300 to 475, and acid number values of from about 0.2 to about 20, preferably about 0.2 to about 10 are given. In the examples only one acid number is disclosed for the polyols (Example 1: 1.4).

4.2.4 In the depolymerisation of polyethylene terephthalate (**PET**), the following molar ratios are given: total amount of polyol (digesting polyol and EG) to total acid (terephthalic acid, **TPA**, and polycarboxylic acid) = 1.5 to 6; PET to polycarboxylic acid = 1.5 to 3.5; digesting polyol to EG = 1.8 to 2.5 (page 8, line 36 to page 9, line 16).

4.2.5 The document does not disclose a polyester resin being the reaction product of the four mandatory components (A) to (D) in their respective amounts as defined in present Claim 1.

4.3 D2 relates to the recycling of polyester resins by depolymerisation in an extruder by means of 0.015 to 0.047 mols of a substance selected from water and a starting compound of the polyester per mol of dicarboxylic acid moieties in the polyester (Claim 1; page 6, lines 19 to 23). The material can subsequently be polymerised with other acids or glycols to new and different polyester resins (page 3, lines 6 to 10). On page 9, last paragraph and on page 10, examples of the various possible components suitable for that process are given. On page 11, lines 16 to 28, a number of co-reactants for possible modifications of PET are listed

which are suitable for the preparation of copolyester resins. However, oxyalkylated polyols are not mentioned. An aqueous medium to dissolve or disperse the resin is not contemplated either.

4.4 D3 relates to a method of manufacturing polyester resins suitable for use in the production of protective coatings, having improved chemical and thermal resistance. The resins are applicable as powders to substrates such as metal surfaces by the fluidisation method, by electrostatic spray or by any other dusting method with improved spreading properties (page 1, lines 8 to 47). According to Claim 1, polyterephthalate resins are subjected to alcoholysis with a glycol and a trihydric alcohol. The product thus obtained has mostly hydroxylic groups on the ends of the particles, and it can be cured at high temperatures with the aid of hardeners and with, if possible, the addition of accelerators. Hardeners mentioned are polytitanates and polyisocyanates (page 2, lines 1 to 8). The citation does not refer to the presence of IA and oxyalkylated polyol nor to specific amounts of the individual components as defined in Claim 1.

4.5 None of the citations disclosing a water soluble or water dispersible polyester resin characterised by a combination of product features and process features within the terms of the application, it is concluded that the subject-matter of Claim 1 is novel (Article 54(1) and (2) EPC).

5. *Inventive step*

5.1 The patent application in suit concerns water soluble or water dispersible polyester resins suitable for use as water-based sizing compositions for fibres, fabrics and paper (page 5, lines 11 to 27). At lines 28 to 35, further particulars of such sizing solutions or dispersions are given.

5.2 None of the citations relates to this particular technological field of sizing agents for fibres, fabrics or paper. The citations show similarity to the subject-matter of the patent application insofar as all of them describe the depolymerisation of polyterephthalate resins.

5.2.1 D1 is the only document mentioning all the components which are used in accordance with Claim 1. Therefore the Board, like the Appellant and the Examining Division, regards D1 as representing the closest state of the art.

5.2.2 As previously indicated, D1 relates to a polyester polyol which is useful in the preparation of cellular foam or non-cellular materials, in particular polyurethane or polyisocyanurate foams. These foams can be obtained by reaction of these polyester polyols and further polyols with a polyisocyanate component (see the description from page 10, line 36 onwards). Polyurethane foams result from an equivalent ratio of essentially 1:1 to 1:1.2, polyisocyanurate foams are obtained with a "minor amount of polyol" (page 11, lines 10 to 23).

5.2.3 Apart from recycling polyalkylene terephthalate scrap and saving energy, the citation aims at improved cellular foams having a combination of advantageous properties, including reduced friability and high thermal stability and compressive strength. Moreover, an improved method of producing these foams should also be defined (page 2, lines 28 and 29; page 3, lines 30 to 35). Sizing properties are not mentioned.

5.3 In accordance with the introductory statements in the application documents, the technical problem underlying the application in suit may be seen in the provision of a material suitable for sizing fibres, fabrics or paper on the basis of waste material which could be recycled in a convenient manner, such as e.g. waste terephthalate plastic material.

5.4 According to the patent application in suit, this problem is solved by a water soluble or water dispersible polyester resin which can be obtained in a two-step process involving depolymerisation of terephthalate polymer by heating with glycols and oxyalkylated polyols followed by modification of the so-obtained intermediate product with isophthalic acid within specific weight percentages.

5.5 According to Examples 1 to 4, water soluble or water dispersible sizing agents are obtained from a number of such terephthalate resins, which improve smoothness and elongation of cellulosic, polyester, polyamide and acrylic fibres and fabrics. Consequently, the various aspects of the above defined technical problem are effectively solved by the product as defined in Claim 1.

6. It remains to be decided whether this solution was obvious to a person skilled in the art having regard to the state of the art relied upon in the decision under appeal.

6.1 It is evident from the above considerations that D1, which is not concerned with textile sizing compositions, cannot provide by itself any information which would give an incentive to consider a modification of the polyester polyol within the terms of the application in suit.

6.2 The same argument applies to D2 and D3 (see points 4.3 and 4.4) which like D1 aim at entirely different applications, *viz.* to provide starting material for solid polymers or curable polymers by depolymerising polyester scrap.

6.3 It follows that the solution according to Claim 1 would not have been obvious to a person skilled in the art having regard to the citations relied upon by the Examining Division, whether considered in isolation or in combination and, therefore, involves an inventive step.

7. In view of the above, the Board has come to the conclusion that Claim 1 meets the requirements of the EPC.

8. Claim 6 concerning fibres, fabric or paper coated with the water-soluble or water-dispersible resin according to Claim 1, and Claims 2 to 5, which relate to preferred embodiments of Claim 1, are supported by the patentability of the main claim and thus also

allowable.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The case is remitted to the first instance with the order to grant a patent on the following basis:

   - Claim 1 as submitted on 8 July 1999,

   - Claims 2 to 6 as submitted on 22 June 1999, after appropriate adaptation of the description.

The Registrar: The Chairman:

E. Görgmaier C. Gérardin