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**D E C I S I O N**  
**of 15 September 2003**

**Case Number:** T 0414/00 - 3.3.3

**Application Number:** 93303506.5

**Publication Number:** 0569234

**IPC:** C08J 9/32

**Language of the proceedings:** EN

**Title of invention:**

Thermoexpandable microcapsules having small particle size and production thereof

**Patentee:**

MATSUMOTO YUSHI-SEIYAKU CO., LTD.

**Opponent:**

Casco Products AB

**Headword:**

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**Relevant legal provisions:**

EPC Art. 56

**Keyword:**

"Inventive step (yes) - problem and solution"

**Decisions cited:**

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**Catchword:**

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Case Number: T 0414/00 - 3.3.3

**D E C I S I O N**  
of the Technical Board of Appeal 3.3.3  
of 15 September 2003

**Appellant:** Casco Products AB  
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**Decision under appeal:** Interlocutory decision of the Opposition  
Division of the European Patent Office posted  
17 February 2000 concerning maintenance of  
European patent No. 0569234 in amended form.

**Composition of the Board:**

**Chairman:** R. Young  
**Members:** A. Däweritz  
E. Dufrasne

## Summary of Facts and Submissions

I. The grant of European patent No. 0 569 234 in respect of European patent application No. 93 303 506.5, filed on 6 May 1993 and claiming priority of 8 May 1992 of an earlier application in Japan (115854/92), was announced on 16 July 1997 (Bulletin 1997/29) on the basis of five claims. The title of the patent reads "Thermoexpandable microcapsules having small particle size and production thereof".

Independent Claims 1, 3 and 5, respectively, read as follows:

- "1. Thermoexpandable microcapsules which comprise a volatile organic solvent in a polymer shell, said microcapsules having an average diameter of 1 - 10  $\mu\text{m}$  and a maximum volumetric expansion rate of 10 times or more characterised by said solvent containing an alcohol and/or a fatty acid each of which contains a saturated or unsaturated and possibly branched,  $\text{C}_8 - \text{C}_{22}$  hydrocarbon chain."
  
- "3. A process for the production of thermoexpandable microcapsules of a particle size of 1 - 10  $\mu\text{m}$ , which comprises suspension-polymerizing a polymerizable monomer in the presence of a volatile organic solvent, characterised in that the polymerization is carried out in the presence of an alcohol and/or a fatty acid each of which contains a saturated or unsaturated and possibly branched  $\text{C}_8 - \text{C}_{22}$  hydrocarbon chain."

"5. Use of thermoexpandable microcapsules as claimed in claim 1 or claim 2 or when prepared by the process of claim 3 or claim 4 in the manufacture of porous, insulation, light-weight, or covering, materials."

Claims 2 and 4 were dependent claims on Claims 1 and 3, respectively.

II. On 14 April 1998, a Notice of Opposition was filed in which revocation of the patent in its entirety was requested on the grounds of lack of novelty in regard to a document according to Article 54(3) EPC and of inventive step within the meaning of Article 56 EPC. These objections were based on eight documents, including

**D1:** EP-A-0 486 080 (published 20 May 1992),

**D3:** US-A-3 615 972,

**D4:** Ullmann's Encyclopedia of Industrial Chemistry, Volume A9, VCH Weinheim, 1987, pages 297 to 323 (copies of pages 313, 316 and 317 were not provided),

**D5:** US-A-5 053 436,

**D6:** US-A-4 786 696, and

**D7:** Kirk-Othmer, Encyclopedia of Chemical Technology, 3<sup>rd</sup> Edition, Volume 8, Wiley-Interscience, New York, 1979, pages 900 to 923.

In reply to the opposition, the Proprietor deleted the reference to the fatty acid from the characterising parts of Claims 1 and 3 (above) in order to meet the novelty objection on the basis of D1 (letter of 1 December 1998).

III. In an interlocutory decision issued in writing on 17 February 2000, the Opposition Division held that the grounds of opposition did not prejudice the maintenance of the patent in the amended form as indicated above. Consequently, the independent claims as maintained read as follows:

- "1. Thermoexpandable microcapsules which comprise a volatile organic solvent in a polymer shell, said microcapsules having an average diameter of 1 - 10  $\mu\text{m}$  and a maximum volumetric expansion rate of 10 times or more characterised by said solvent containing an alcohol which contains a saturated or unsaturated and possibly branched,  $\text{C}_8 - \text{C}_{22}$  hydrocarbon chain."
  
- "3. A process for the production of thermoexpandable microcapsules of a particle size of 1 - 10  $\mu\text{m}$ , which comprises suspension-polymerizing a polymerizable monomer in the presence of a volatile organic solvent, characterised in that the polymerization is carried out in the presence of an alcohol which contains a saturated or unsaturated and possibly branched  $\text{C}_8 - \text{C}_{22}$  hydrocarbon chain."

"5. Use of thermoexpandable microcapsules as claimed in claim 1 or claim 2 or when prepared by the process of claim 3 or claim 4 in the manufacture of porous, insulation, light-weight, or covering, materials."

(a) In the decision, it was established that novelty of the claims as amended had no longer been disputed by the Opponent.

(b) As regards inventive step, D3 was identified as representing the closest state of the art, which disclosed all the features of the preamble of Claim 1 of the patent in suit as amended. In Example 49, the thermoexpandable microcapsules (beads) expanded upon heating about five times their original diameter, which corresponded to the maximum expansion rate of 10 times or more as required in Claim 1 of the patent in suit.

However, the document did not disclose that an alcohol having 8 to 22 carbon atoms was to be used in the polymerisation process of Claim 3 and to be contained in the said volatile organic liquid encapsulated in the thermoexpandable microcapsules according to Claim 1.

(c) The Opposition Division further relied on a statement of the Proprietor in its letter dated 1 December 1998 (page 2, last paragraph) that when repeating Example 49 of D3 by preparing beads having an average particle size of about 5  $\mu\text{m}$  (determined by laser diffraction), expanding the beads obtained at several temperatures between 100

and 140°C, and determining the particle size after expansion, most particles had not expanded, though some beads were observed after the expansion which showed diameters of 25 µm.

Accordingly, the description "the beads expanded about five times their original diameters" would not mean that this had been the average expansion rate of the whole of the expandable beads. If the particle size was determined by microscope such a selection was possible.

- (d) In view of the fact that the above results had not been refuted by the Opponent, the technical problem to be overcome was, consequently, not seen only as the provision of further thermoexpandable microcapsules having a small particle size and high expansion rate and a further process for preparing such microcapsules, as would have appeared from the text of the said example in D3, but, more precisely, as "the provision of thermoexpandable microcapsules having a small size which expand more homogeneously with a high expansion rate than the particles of said example and as the provision of a method of preparation of said microcapsules" (decision under appeal: page 3, third complete paragraph; page 4, first paragraph).
- (e) Document D3 did not contain any hint that this problem could be solved by polymerising the monomers in the presence of an alcohol as defined in the claims of the patent in suit.

(f) A further argument raised by the Opponent was not accepted in the decision: it was based on the fact that the polymerisation was carried out in D3 in the presence of (1) a water-dispersible solid colloid and (2) a water-soluble "promotor" and/or (3) an electrolyte and/or (4) a colloid-active modifier such as peptizing agents or surface-active agents, and that such agents, known from D4 to D7, included C<sub>8</sub> - C<sub>22</sub> alcohols.

(g) Since the latter documents did not, however, deal with the preparation of expandable microspheres, there was no hint to the skilled person that by selecting specifically those alcohols as defined in the above claims the above relevant technical problem (section III.(d), above) could be overcome. No way was seen how the skilled person could have combined the teaching of D3 with the teachings of these other documents in order to solve the relevant problem.

Consequently, an inventive step was acknowledged.

IV. On 17 April 2000, a Notice of Appeal was lodged by the Opponent (Appellant) against this decision with simultaneous payment of the prescribed fee. The Appellant requested that the decision be set aside and the patent in suit be revoked in its entirety.

In the Statement of Grounds of Appeal, filed on 15 June 2000, the Appellant maintained its previous objection of lack of inventive step.



- (a) To that end, it relied on D3 as closest available prior art and argued that this prior art had disclosed all the features in the preamble of Claim 1. The maximum volumetric expansion rate was also rated as high, eg in numbers of  $5^3 = 125$  (Example 49). The polymerisation was carried out by dispersing a polymerisable liquid in a nonsolvent aqueous medium comprising water and, *inter alia*, a colloid-active modifier such as surface-active agents (column 5, lines 48 to 64), the latter term being equivalent to "surfactant" and "emulsifier".
- (b) It had been well known to use alcohols which contained saturated or unsaturated and possibly branched  $C_8 - C_{22}$  hydrocarbon chains as surfactants (emulsifiers). Thus, in D7, reference was made to propylene glycol monolaurate, sorbitan monotearate and monolaurate, glycerol monostearate and monolaurate. These compounds were considered as alcohols according to the definition in Claim 1. Other examples of this kind serving for the same purpose, including suitable amounts in which they could be used, were to be found in D5 and D6, in particular sorbitan monooleate, ethoxylated oleyl alcohol and ethoxylated lauryl alcohol.
- (c) On the basis of a single experiment by the Patent Proprietor (Respondent), the result of which had not been as good as that reported in the said Example 49 of D3, the technical problem to be overcome could not be redefined as had been done by the Opposition Division, because no support was provided therefor in the patent in suit. In

particular, there was no disclosure, nor teaching, nor indication in the patent in suit about 'homogeneous expansion' or similar expression. Instead, the patent addressed on the combination of small particle sizes of the beads and a high maximum volumetric expansion rate (Statement of Grounds of Appeal, the paragraph bridging pages 2 and 3). Consequently, the problem underlying the patent in suit lay in the provision of further thermoexpandable microcapsules having a small particle size and high expansion rate and a further process for preparing such microcapsules.

- (d) As regards the solution of this latter technical problem, it would have been clearly obvious to the person skilled in the art to arrive at the subject-matter defined in Claim 1 or Claim 3 by combining the teachings of D3 and D7 or D5/D6.

Since, on the one hand, it had belonged to the common general knowledge that surfactants (emulsifiers) reduced the energy required for emulsification to obtain small droplets of the disperse phase (D4), there had been an incentive to the skilled person to use a surfactant (emulsifier) in the process of D3.

On the other, it had been obvious "that a higher amount of surfactant/emulsifier (as used with octanol) would reduce the energy required for emulsification to obtain small particles and/or result in smaller particles when using the same amount of energy for emulsification" (page 4 of the Statement of Grounds of Appeal).

Therefore, no inventive step was involved in selecting an alcohol of the kind defined in Claim 1 for this purpose.

V. In its reply dated 20 October 2000, the Respondent (Proprietor) supported the findings in the interlocutory decision under appeal. It was well established case law that the specific problem in the objective assessment of inventive step could be restated when prior art came to light which had not been considered in the original patent application.

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.

### **Reasons for the Decision**

1. The appeal is admissible.

2. *Admissibility of amendments*

The claims as granted were amended during the opposition proceedings by deletion of the phrase "and/or fatty acid each of" from Claims 1 and 3. The deletion of one of two independent alternatives in the claim does not result in added subject-matter but in a further limitation of the claims. Consequently the requirements of Article 123(2) and (3) EPC are fulfilled. The allowability of the amendments was in any case not challenged by the Appellant.

3. *Novelty*

Novelty of the subject-matter in the patent in suit as amended was no longer contested in the appeal proceedings. Consequently, the Board holds the claimed subject-matter to be novel.

4. *Problem and solution*

The patent in suit concerns thermoexpandable microcapsules having a small particle size, their production and use. In particular, it concerns thermoexpandable microcapsules which comprise a volatile organic solvent in a polymer shell, the microcapsules having an average diameter of 1 to 10  $\mu\text{m}$  and a maximum volumetric expansion rate of 10 times or more when heated.

4.1 Such microcapsules are known from document D3, which by common consent represents the closest state of the art.

4.2 According to D3, there is disclosed a method of preparing a hollow monocellular particle comprising a thermoplastic expandable synthetic resinous polymeric particle having a generally spherical shape and having encapsulated therein generally concentrically as a spherical occlusion, a distinct and separate liquid phase consisting essentially of a volatile organic liquid raising agent (ie blowing agent), the liquid becoming gaseous at a temperature below the thermoplastic temperature of the particle, the particle being generally impermeable to the raising agent (blowing agent), heating the particle to a temperature

sufficient to cause heat plasticization of the polymer shell and volatilization of the raising agent thereby expanding the particle to form a monocellular hollow generally spherical shell having a gaseous centre and cooling the particle to a temperature below the thermoplastic temperature thereof (Claim 1).

The preparation of the above product includes the provision of an aqueous dispersion of (1) organic monomeric material suitable for polymerization to a thermoplastic material having the desired physical properties, (2) a liquid raising or blowing agent which exerts little solvent action on the resulting polymer, and (3) a dispersion stabilizing material which is utilized to maintain the dispersion, followed by polymerization of the monomeric material to the beads described above (column 3, lines 11 to 25). Typical blowing or raising agents are aliphatic hydrocarbons such as ethane, propane, neopentane and hexane (column 4, lines 9 to 17). The diameter of the particles may be from 0.5  $\mu\text{m}$  to about 0.5 cm, in particular, 1 to 50  $\mu\text{m}$ , preferably 2 to 10  $\mu\text{m}$  (Claims 47, 48 and column 5, lines 30 to 34).

According to Example 49 the product obtained by copolymerization of styrene and methacrylic acid in the presence of neopentane is described as a plurality of small beads having a diameter of about 5  $\mu\text{m}$  which expanded to about 5 times their original diameters upon heating to 130°C.

- 4.3 Whilst it is apparent from the above that Example 49 of D3 discloses, on the face of it, a microcapsule having a diameter within the range of 1 to 10  $\mu\text{m}$  and a maximum volumetric expansion rate of 10 times or more as required by Claim 1 of the patent in suit, and therefore differs from the claimed subject-matter only by the presence, in the solvent, according to the patent in suit, of an alcohol which contains a saturated or unsaturated and possibly branched  $\text{C}_8\text{-C}_{22}$  hydrocarbon chain (which was not disputed by the parties), the question arises, and forms the principal issue in this appeal, as to the appropriate form of the technical problem objectively arising, in relation to the claimed subject-matter, from the above disclosure, in particular, Example 49 thereof.
- 4.4 In particular the question arises as to whether the technical problem was to be seen as (i) "the provision of thermoexpandable microcapsules having a small size which expand more homogeneously with a high expansion rate than the particles of said example and as the provision of a method of preparation of said microcapsules" as determined by the Opposition Division, or (ii) merely as "the provision of further thermoexpandable microcapsules having a small particle size and high expansion rate and a further process for preparing such microcapsules", as canvassed by Appellant (cf section III(d); section IV(c), above).
- 4.5 This question boils down to whether the Opposition Division was justified in relying upon the statement of the Respondent in its letter dated 1 December 1998 (page 2, last paragraph), that when repeating Example 49 of D3 by preparing beads having an average

particle size of about 5  $\mu\text{m}$ , expanding the beads obtained at several temperatures between 100 and 140°C, and determining the particle size after expansion, most particles had not expanded (cf section III(c), above). This statement formed the basis of the more sophisticated formulation (i) compared with the more primitive formulation (ii).

4.6 In the Board's view, the statement in the letter of the Respondent dated 1 December 1998 relied upon by the Opposition Division amounts to a report of experiments actually carried out and thus as evidence relevant to the assessment of the objective technical performance of the methods disclosed in the prior art as compared with the subject-matter of the patent in suit.

4.6.1 Whilst criticising that formulation (i) was based on a single experiment, in which "The Proprietor apparently did not obtain as good a result as the result stated in Example 49", and on the conclusion (on the basis of that experiment) "that the result of Example 49 of D3, ie 'the beads expanded about five times their original diameter', should not be interpreted as stated in D3" (Statement of Grounds of Appeal, page 2, paragraphs 5 and 6, section IV.(c), above), the Appellant has failed to recognise what in the Board's view is a fundamental difference in emphasis between the disclosure of the patent in suit and that of D3.

4.6.2 Firstly, it is conspicuous to the Board that the prior art acknowledged in the patent in suit is JP-B-42-26524, which is based on the same prior US patent applications as D3 (US Serial Nos. 246529 and 306050 of 21 December 1962 and 3 September 1963, respectively).

4.6.3 Furthermore, in relation to the problem of expandability of small microcapsules, it is explicitly stated in this acknowledgement of prior art that, when the particle size is controlled to be smaller than 10  $\mu\text{m}$ , thermoexpandability is extremely reduced, or only microcapsules having a very small expansion rate or little expansion are obtained (column 1, lines 21 to 23 and 25 to 27). In particular, in relation to such smaller particle sizes, it is set forth that, "During suspension polymerization many polymerized fine particles not containing the expanding agent may be formed. This is considered to be one reason for the reduction of the thermoexpandability." (column 1, lines 38 to 42). Thus, it is clear that the patent in suit is directly concerned with the problem of inhomogeneous expansion of microcapsules of fine particle size corresponding to the parameter range given in Claim 1 of the patent in suit.

4.6.4 This represents, in the Board's view and contrary to the submission of the Appellant, a clear concern in the patent in suit with forming more homogeneous thermo-expandable microcapsules than the prior art, in particular as represented by D3. This concern is further evidenced in the wording of Claim 1, which refers to the need for thermoexpandable microcapsules having a particular average size, whilst the



corresponding disclosure of Example 49 in D3 merely refers to a "plurality of small beads".

Nor has the Appellant provided any counter evidence, for instance in the form of experiments of its own, which could refute or relativate the evidence of the Respondent (cf section III(c), above).

4.6.5 Consequently, the Board sees no reason to refrain from taking due account of this evidence in assessing the technical contribution provided by the patent in suit, or, therefore, to fail to concur with the adoption, in the decision under appeal, of the corresponding formulation (i) of the relevant technical problem objectively arising, in its assessment of inventive step. Nor is the objection of the Appellant that there was no support in the patent in suit for the formulation (i) convincing in the light of the details referred to above with respect to D3 and its Japanese counterpart, respectively.

4.6.6 In summary, the Board finds that the Appellant has failed to show that the decision under appeal was incorrect in arriving at its statement of the technical problem objectively arising from the disclosure of D3 corresponding to formulation (i), above.

4.7 Since, furthermore, the reasoning given in the decision under appeal in relation to the assessment of inventive step on the basis of the above formulation (i) of the technical problem has not been challenged by the Appellant in any respect, the Board sees no reason to come to a different conclusion, in relation to the subject-matter claimed in the patent in suit, than did the decision under appeal.

## **Order**

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

The appeal is dismissed.

The Registrar:

The Chairman:

E. Görgmaier

R. Young