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D E C I S I O N
of 11 February 2004

Case Number: T 0998/01 - 3.3.5

Application Number: 92309548.3

Publication Number: 0539142

IPC: B01J 13/02

Language of the proceedings: EN

Title of invention:

Microcapsules containing inorganic colloids and method for making the same

Patentee:

MINNESOTA MINING AND MANUFACTURING COMPANY

Opponent:

BASF Aktiengesellschaft Patente, Marken und Lizenzen

Headword:

Microcapsules/3M

Relevant legal provisions:

EPC Art. 54(1) and 56

Keyword:

"Novelty - yes; inventive step - yes, non-obvious alternative"

Decisions cited:

-

Catchword:

-



Case Number: T 0998/01 - 3.3.5

D E C I S I O N
of the Technical Board of Appeal 3.3.5
of 11 February 2004

Appellant:
(Opponent)

BASF Aktiengesellschaft
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D-67056 Ludwigshafen (DE)

Representative:

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Respondent:
(Proprietor of the patent)

MINNESOTA MINING AND MANUFACTURING COMPANY
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Representative:

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Decision under appeal:

Decision of the Opposition Division of the
European Patent Office posted 5 July 2001
rejecting the opposition filed against European
patent No. 0539142 pursuant to Article 102(2)
EPC.

Composition of the Board:

Chairman: M. M. Eberhard
Members: G. J. Wassenaar
H. Preglau

Summary of Facts and Submissions

- I. European patent No. 0 539 142 was opposed by the appellant (opponent). The patent comprised 16 claims of which claim 1 read as follows:

"Microcapsules having a 50% volumetric diameter ranging between 3 to 12 micrometers, said microcapsules comprising an oleophilic fill material retained within a synthetic thermoset polymer shell, said shell further comprising colloidal inorganic particles selected from silica particles and zirconium dioxide particles, said particles having average diameter of less than 0.03 micrometers and having a surface energy selected such that during manufacture of the microcapsule from a solution having an oil phase and an aqueous phase, the particle will migrate to the interface of the oil phase and the aqueous phase."

- II. The opposition grounds were lack of novelty and lack of inventive step. The arguments were supported by the following documents:

D1: US-A-3 615 972

D2: Data sheet "LUDOX® COLLOIDAL SILICA"

D3: EP-B-0 026 914.

- III. The opposition division rejected the opposition. With respect to novelty it was held that D1 does not disclose microcapsules having a synthetic thermoset polymer shell.

With respect to inventive step it was held that D3 represented the closest prior art and that it was not obvious to replace the polyelectrolyte used in D3 with the colloidal silica mentioned in D1.

IV. The appellant lodged an appeal against the decision of the opposition division to reject the opposition. In the grounds of appeal it was again argued that the subject-matter of claim 1 as granted lacked novelty and did not involve an inventive step. The following two further prior art documents were cited to support the arguments:

D4: C A Finch, "Polymers for microcapsule walls",
Chemistry and Industry, 1985, pages 752 to 756,

D5: Kondo, Asali "Microcapsule Processing &
Technology", J. Wade, LC 79-18821, pages 46 to 58.

Oral proceedings took place on 11 February 2004.

V. The arguments of the appellant can be summarized as follows:

Lack of novelty

D1 disclosed microcapsules fulfilling all the requirements of present claim 1. Although D1 was directed to microcapsules having an expansible thermoplastic polymer shell, it also disclosed cross-linked non-thermoplastic resin as shell material. Such a cross-linked resin was a synthetic thermoset polymer within the meaning of present claim 1.

Lack of inventive step

Starting from D1 as the closest prior art, the problem to be solved was to reduce the permeability of the polymer shell. D1 itself already taught that the permeability might be reduced by cross-linking the polymer. D4, a review article, also disclosed that the permeability could be reduced by cross-linking. It was thus obvious to solve said problem by curing the polymer used in D1 to such an extent that a cross-linked, non-thermoplastic resin was obtained.

Starting from D3, the problem to be solved was to provide alternative microcapsules having a small uniform particle size and low permeability. It was obvious to solve this problem by replacing the dispersion aid used in D3 by another known dispersion aid. Colloidal silica, mentioned in D1, D4 and D5 as additive in the preparation of microcapsules in an aqueous medium, was such a dispersion aid. Since D1 further disclosed that colloidal silica was particularly suitable as dispersant for stabilizing the polymerizable liquid droplets, it was also obvious to use the same kind of colloidal silica as used in the examples of D1 as dispersant in a method according to D3, thereby obtaining microcapsules as now claimed.

Starting from D3 it was also an obvious alternative to replace the shell forming polymer system by the system disclosed in D1, comprising colloidal silica as dispersant, and cross-linking the polymer to a thermoset polymer.

VI. The respondent (patentee) refuted the arguments of the appellant and submitted four sets of claims as auxiliary requests. The arguments of the respondent can be summarized as follows.

Novelty

D1 was directed to expansible microcapsules which required that the polymer was thermoplastic. Although D1 disclosed that the polymer might be cross-linked to a certain extent, it comprised a warning not to cross-link the polymer to such an extent that it would lose its thermoplastic property. This warning could not be regarded as a disclosure of microcapsules comprising a shell of a thermoset polymer. Moreover the composition of such a hypothetical microcapsule was not disclosed. There was certainly no disclosure that such a hypothetical microcapsule would comprise colloidal silica as required by present claim 1. Colloidal silica was only one example of possible dispersants mentioned in D1.

Inventive step

D1 was not an appropriate starting point for an inventive step evaluation, because it related to foaming agents, which belonged to a technical field completely different from that of carbonless copying paper, the field of application of the patent in suit. D3 related to the same technical field and discussed the same essential problems in this field, i.e. to obtain small microcapsules having a narrow size distribution and a low permeability.

Starting from D3 the problem was to provide further microcapsules of comparable size and permeability. It was not obvious to look for a solution to that problem in documents related to a completely different technical field such as D1. The microcapsules of D1, having an expansible thermoplastic shell, were not compatible with microcapsules according to D3, having a pressure sensitive shell of a thermosetting polymer. The dispersion agent used in D1, a water-insoluble solid colloid, was also structurally completely different from the water-soluble polymer having sulfonic acid groups used in D3. Moreover there was no indication in D1 that the colloidal silica used therein would solve the problem. In particular there was no indication that it would reduce the permeability to the same extent as the said soluble polymer used in D3. A skilled person would, in fact, have expected an increase in permeability by using colloidal silica because of its particulate structure. Also D4 did not suggest the replacement of the said water-soluble polymer with a water-insoluble colloid such as colloidal silica. The latter should be considered as a kind of filler, which, according to Table 1 of D4, would increase the permeability. Microcapsules as claimed in the patent in suit, therefore, did not follow in an obvious manner from the cited prior art documents.

VII. The appellant requested that the decision under appeal be set aside and the patent be revoked.

The respondent requested that the appeal be dismissed and the patent be maintained as granted (main request), or that the decision under appeal be set aside and that

the patent be maintained on the basis of one of the four auxiliary requests, filed before the first instance by letter dated 15 June 2000.

Reasons for the Decision

1. Novelty has been attacked on the basis of D1. This document discloses expansible thermoplastic microspheres containing a liquid blowing agent. The microspheres have preferably a diameter between 2 and 10 μm (claim 1, column 11, lines 23 to 26 and example 1). The blowing agent may be aliphatic hydrocarbons, chlorofluorocarbons and tetra alkyl silanes, which are generally oleophilic substances (column 4, lines 9 to 37). The oleophilic blowing agent used in example 1 is neopentane. During preparation a dispersing agent is used in the form of a water-dispersible, water-insoluble solid colloid (column 4, lines 38 to 44, and column 5, lines 48 to 71). In the examples a colloidal silica dispersion in water, available under the trade name of "Ludox HS", has been used (example 1). It is uncontested that the colloidal particles in "Ludox HS" are silica particles within the meaning of claim 1 as granted and remain in the shell of the microspheres. The monomer composition from which the thermoplastic shell is made may comprise a cross-linking agent, which serves to increase the flow viscosity of the polymeric composition at temperatures sufficiently high to cause volatilization of the blowing agent and subsequent deformation of the originally formed sphere into a larger hollow sphere (column 12, lines 48 to 55).

2. The microcapsules according to claim 1 as granted differ from those disclosed in D1 in that the shell is made of a thermoset polymer. The appellant's view that D1 also discloses microcapsules comprising a shell of a thermoset polymer cannot be shared. This view is based on the sentence: "If the polymerization conditions are such that a cross-linked non-thermoplastic resin is prepared there can be little or no expansion." (column 13, lines 1 to 3). In the board's view this sentence simply expresses the well known fact that cross-linking reduces the thermoplastic properties of a polymer, and that, in order to obtain expansible microspheres, which is the object of the process disclosed in D1, the resin should not be cross-linked to such an extent that it loses its thermoplastic properties. It is thus a warning to avoid certain polymerization conditions and not a disclosure of microcapsules having a thermoset polymer shell, let alone of microcapsules having in combination all the properties required by present claim 1. Thus D1 does not destroy the novelty of claim 1 as granted.
3. Since D1 relates to expansible microspheres having a thermoplastic shell, which is totally contrary to the teaching of the patent in suit to provide pressure sensitive microspheres having a synthetic thermoset polymer shell, D1 cannot be regarded as the closest prior art in an inventive step evaluation. But even if, for the sake of argument, D1 is considered as the closest prior, the appellant's arguments with respect to lack of inventive step are not convincing for the following reasons.

4. According to the appellant, starting from D1, the problem to be solved would have been a reduction of the permeability of the polymer shell. D1 teaches indeed that diffusion of the blowing agent through the particle wall may be a problem in certain circumstances and that for high proportions of blowing agent it is often desirable to employ a monomer system which on polymerization results in a cross-linked polymer shell (column 17, line 72 to column 18, line 22). The board does not dispute that the skilled person might have derived therefrom that the permeability could be reduced by cross-linking the polymer. D1 also mentions some polymer systems comprising difunctional monomers for cross-linking (column 18, lines 22 to 32). This passage in D1 is followed by the observations that contrary to the normal expectations, such copolymers are generally not rigidly cross-linked, and on heating are capable of expansion but that they usually do not expand to the same degree as a material without the difunctional monomer (column 18, lines 33 to 37). Thus following the teaching of D1, the skilled person would consider a polymer system comprising a difunctional monomer in order to reduce the permeability but, in view of the warning in column 13, lines 1 to 3 of D1, certainly not a polymer system and conditions which result in a non-thermoplastic resin, which cannot be expanded. Using a polymer system and conditions which result in a non-expansible thermoset polymer shell would be contradictory to the whole teaching of D1. The average skilled person would normally follow the general teaching of a document; ignoring essential features disclosed therein and acting against the general teaching thereof, are rather an indication of an inventive step.

5. D4 also discloses that cross-linking reduces the permeability of polymers used in capsule walls (page 753, Table 1). D4 makes further reference to a polymer system of the type as used in D1, without mentioning D1 (page 754, Table 5). D5 discloses in Table 6.1 the same polymer system with an explicit reference to a Japanese patent which would correspond to D1. It is thus obvious to combine the teachings of documents D1, D4 and D5. By such a combination the skilled person would however not arrive at microcapsules having a thermoset polymer shell for the reasons given at the end of the above paragraph.

6. In agreement with the position taken in the contested decision and the submissions made by the respondent, the board considers that D3 represents the closest prior art. This document discloses microcapsules comprising a thermoset polymer shell for use in pressure sensitive carbonless copying paper. The polymer shell is formed by condensing melamine-formaldehyde precondensates and/or their C₁ - C₄ alkyl ethers in water in which a substantially water-insoluble fill material is dispersed in the presence of a water-soluble polymer comprising sulfonic acid groups (claims 1, 10, 11). The obtained microcapsules have a narrow size distribution within the range of 1 to 8 µm and low permeability (low JCF number); see page 3, lines 57 to 65 and the examples. The microcapsules obtained by the examples of the patent in suit also have a narrow size distribution and low permeability but the respondent has not provided evidence for any improvement of the properties of the microcapsules of the patent in suit compared with those obtained

according to D3. Thus, in agreement with the reasons in the contested decision, the problem underlying the invention is to provide further microcapsules suitable for use in carbonless copying paper and having size and permeability characteristics comparable to those obtained according to D3.

The respondent proposes to solve this problem by providing microcapsules as defined in claim 1 as granted having a shell comprising colloidal silica or zirconium dioxide particles.

It is uncontested that the microcapsules according to claim 1 have a size and permeability in the same order of magnitude as the microcapsules obtained according to D3 so that it is credible that the claimed microcapsules actually solve the said problem.

7. D3 itself does not provide a hint that dispersant other than water-soluble polymers containing sulfonic acid groups could be used to solve that problem. On the contrary, it is shown in D3 that even a small change in the nature of the dispersant, such as the use of polymers containing carboxylic groups instead of sulfonic acid groups, provides inferior products (see comparative examples, pages 14 to 15).
8. D4 discloses that for micro encapsulation by in-situ polymerisation certain additives are used. Amongst these additives colloidal silica is mentioned (Table 5). The effect of the additives is not disclosed. There is no indication that with colloidal silica as additive microcapsules could be obtained having a low permeability. Furthermore, D4 discloses that the presence of a filler in the polymer shell possibly increases the permeability (see Table 1). Since

colloidal silica consists of small insoluble particles, the skilled person would fear that it could act as a filler and therefore would not try it if microcapsules with a low permeability were to be prepared.

9. D1 proposes among other water-insoluble solid colloids the use of colloidal silica of the same kind as used in the patent in suit as dispersion aid. It is indicated that they function as a stabilizer for the limited coalescence of the small polymerizable liquid droplets, but nothing is disclosed about their impact upon the permeability of the shell after polymerization (column 4, lines 38 to 44; column 5, line 48 to column 6, line 30; column 7, line 25 to column 8, line 9 and example 1). It was thus not obvious that colloidal silica would solve the above mentioned problem. Moreover, a skilled person, starting from D3, would not pay much attention to D1 because it not only relates to the rather remote art of foaming particles, but uses an essentially different polymerization system. According to D3 the polymer of the shell is formed by condensation of precondensates dissolved in the aqueous phase, in the presence of the polymer containing the sulfonic acid groups, also dissolved in water (claim 1 and examples), whereas according to D1 both the monomers and the blowing agent are in the oil phase and polymerization is performed in the presence of benzoyl peroxide or potassium dichromate as a catalyst (column 4, lines 60 to 68 and the examples). The skilled person would therefore not expect to find a solution of the problem in D1. The skilled person would also be reluctant to replace the water-soluble dispersant from D3 by a particulate water-insoluble

dispersant such as colloidal silica for the reasons set out under point 8 above.

10. In view of said differences in the technical field and polymerization system it is also not obvious to replace the polymerization system of D3 by a polymerization system as used in D1 and to cross-link the thermoplastic polymer to such an extent that the polymer shell becomes a thermoset polymer.
11. The other documents on file do not comprise any incentive for the skilled person to replace the sulfonic acid groups containing polymer of D3 with colloidal inorganic particles according to present claim 1. Since the appellant did no longer rely on these documents in the appeal proceedings there is no need to discuss them here. It follows from the above that the subject-matter of claim 1 cannot be derived in an obvious manner from the prior art as disclosed in the documents on file.
12. Claim 8 is directed to a sheet material containing the microcapsules of any preceding claim. The further independent claim 9 concerns a process for producing microcapsules comprising all the features of claim 1. The reasons for inventive step therefore equally apply to claims 8 and 9. Claims 1, 8 and 9 being allowable, the same applies to dependent claims 2 to 7 and 10 to 16, whose patentability is supported by that of claims 1 and 9.

Order

For these reasons it is decided that:

The appeal is dismissed.

The Registrar:

The Chairman:

A. Wallrodt

M. M. Eberhard