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Datasheet for the decision
of 16 July 2015

Case Number: T 0768/14 - 3.3.07
Application Number: 01931125.7
Publication Number: 1272159


Language of the proceedings: EN

Title of invention:
STABLE AQUEOUS SURFACTANT COMPOSITIONS

Patent Proprietor:
Lubrizol Advanced Materials, Inc.

Opponents:
HENKEL AG & CO. KGAA
Rohm and Haas Company
BASF Personal Care and Nutrition GmbH

Headword:
STABLE AQUEOUS SURFACTANT COMPOSITIONS/Lubrizol Advanced Materials, Inc.

Relevant legal provisions:
EPC Art. 56

Keyword:
Inventive step - (no)
Decisions cited:

Catchword:
DECISION
of Technical Board of Appeal 3.3.07
of 16 July 2015

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Decision under appeal: Interlocutory decision of the Opposition

Composition of the Board:

Chairman       J. Riolo
Members:       D. Boulois
               D. T. Keeling
Summary of Facts and Submissions

I. European Patent No. 1 272 159 based on application No. 01931125.7 was granted on the basis of a set of 18 claims.

II. Three oppositions were filed under Article 100 (a), (b) and (c) EPC on the grounds that its subject-matter lacked inventive step and extended beyond the content of the application as filed, and that the patent was not sufficiently disclosed.

III. The documents cited during the opposition proceedings included the following:

D1: US-A-4 529 773
D3: GB-A-870 994

IV. The appeal by opponent 2 lies from the decision of the opposition division to maintain the patent as amended. The decision was based on the set of claims of the main request presented at oral proceedings on 27 November 2013, which corresponded to those filed as auxiliary request 3 with the letter of 22 October 2013.

Independent claim 1 of that request read as follows:

"1. A stable, aqueous surfactant containing composition, comprising:
a. at least one anionic, zwitterionic, amphoteric, nonionic, or cationic surfactant, or combinations thereof;

b. at least one substantially crosslinked alkali-swellable acrylate copolymer rheology modifier polymerized from
(i) 20% to 80% by weight of acrylic acid or methacrylic acid, or combinations thereof;  
(ii) 80% to 15% by weight of ethylacrylate, or methylacrylate, or combinations thereof, and  
(iii) from 0.01% to 5% by weight of polyalkeny1 ethers of sucrose or polyalcohols; or trimethylolpropane tri(meth)acrylate, glycidyl methacrylate, N-methylolacrylamide, or combinations thereof

c. water;

wherein said composition is obtained by adding an effective amount of at least one alkaline material to yield a composition having a pH of from 5 to 14; and subsequently adding an effective amount of an acidic material to yield a composition having a reduced pH of from 1 to 12, whereby the pH is reduced by at least 0.5 pH unit,

wherein said acidic material is citric acid, acetic acid, beta-hydroxy acid, salicylic acid, alpha-hydroxy acid, lactic acid, glycolic acid, hydrochloric acid, sulfuric acid, nitric acid, sulfamic acid, phosphoric acid, or natural fruit acids, or combinations thereof, and

wherein the amount of said copolymer is from 0.1% to 10% by weight based upon the total weight of said stable composition, and wherein the amount of said surfactant is from 1% to 80% by weight based upon the total weight of said stable composition."
According to the decision under appeal, D1 was the closest prior art. The question of whether the viscosity-increasing effect attributed to back-acidification according to D1 extended also to non-hydrophobically modified (ASE) copolymers (as disclosed in the patent), or was limited to copolymers comprising ether groups capped with a long hydrophobic chain (HASE copolymers), was irrelevant, since all of the copolymers of D1 differed from those of claim 1 at least in that none of the former were cross-linked. Furthermore, whether comparative or not, ASE copolymers were employed in D1 and displayed viscosities similar to those of the preferred HASE copolymers. The experimental data filed by the proprietor demonstrated the alleged effect (of effective stabilisation, or improved yield value) with respect to the compositions exemplified in D1. The objective technical problem was the provision of an aqueous surfactant-containing composition with improved yield value. In order to solve the problem posed, the skilled person would not have combined the teaching of D1 with that of D6 in order to arrive at claim 1, despite the fact that the latter comprised a statement that the cross-linking of copolymers resulted in suspension stabilisation, since he would not have considered this teaching to be generally valid. The requirements of Article 56 EPC were thus fulfilled. Starting at D3 as the closest prior art would not have resulted in a different conclusion.

V. Opponent 2 (appellant), Rohm and Haas Company, filed an appeal against that decision.

Opponents 01 and 03 were party as of right.
VI. With the reply to the statement setting out the grounds of appeal, the respondent Lubrizol Advanced Materials, Inc. (patent proprietor) filed a new main request and auxiliary requests 1-15.

VII. With letter of 28 April 2015 the appellant submitted further arguments in particular concerning the newly filed claim requests.

VIII. With letter of 7 June 2015 the respondent submitted a new main request and auxiliary requests 1-11 to replace all requests filed previously.

IX. In a communication dated 5 June 2015 sent in preparation of oral proceedings, the Board provided a preliminary opinion on some of the relevant issues.

X. Oral proceedings took place on 16 July 2015, in the absence of opponent 03.

Independent claim 1 of the main request reads as follows, the differences with respect to the main request underlying the contested decision being indicated by **bold** (addition) or strike through (deletion):

"1. A stable, aqueous surfactant containing composition, comprising:

a. at least one anionic, zwitterionic, amphoteric, nonionic, or cationic surfactant, or combinations thereof;

b. at least one substantially crosslinked alkali-swellable acrylate copolymer rheology modifier polymerized from
(i) 20% to 80% by weight of acrylic acid or methacrylic acid, or combinations thereof;
(ii) 80% to 15% by weight of ethylacrylate, or methacrylate, or combinations thereof, and
(iii) from 0.01% to 5% by weight of at least one of polyalkenyl ethers of sucrose or polyalcohols; or trimethylolpropane tri(meth)acrylate, glycidyl methacrylate, N-methylolacrylamide, or combinations thereof

c. water;

wherein said composition is obtained by adding an effective amount of at least one alkaline material to yield a composition having a pH of from 5 to 14; and subsequently adding an effective amount of an acidic material to yield a composition having a reduced pH of from 1 to 12, whereby the pH is reduced by at least 0.5 pH unit,

wherein said acidic material is citric acid, acetic acid, beta-hydroxy acid, salicylic acid, alpha-hydroxy acid, lactic acid, glycolic acid, hydrochloric acid, sulfuric acid, nitric acid, sulfamic acid, phosphoric acid, or natural fruit acids, or combinations thereof, and

wherein the amount of said coploymer is from 0.1% to 10% by weight based upon the total weight of said stable composition, and wherein the amount of said surfactant is from 1% to 80% by weight based upon the total weight of said stable composition."

Claim 1 of auxiliary request 1 differs from that of the main request in that it further specifies the following:

"...and in which
- the total amount of carboxylic acid monomers comprising acrylic acid, methacrylic acid, or combinations thereof is 20% to 80% by weight, and
- the total amount of α,β-ethylenically unsaturated monomers CH₂=CHXY, where X is H or -CH₃ and Y is -COOR, and R is C₁-C₁₀ alkyl is 80% to 15% by weight, and
- the total amount of polyunsaturated compounds selected from the polyalkenyl ethers of sucrose, or polyalcohols; diallylphthalates, divinyl benzene, allyl (meth)acrylate, ethylene glycol di(meth)acrylate, methylene bisacrylamide, trimethylolpropane tri(meth)acrylate, diallyl itaconate, diallyl fumarate, diallyl maleate, zinc (meth)acrylate, glycidyl methacrylate, or N-methylol acrylamide is from 0.01 to 5% by weight."

Claim 1 of auxiliary requests 2 and 3 differ from those of the main request and auxiliary request 1 respectively in that the wording "polymerized from" in feature b. has been replaced by "polymerized from the three components (i), (ii) and (iii)".

Claim 1 of auxiliary requests 4 and 5 differ from those of the main request and auxiliary request 1 respectively in that the wording "polymerized from" in feature b. has been replaced by "polymerized from a monomer composition consisting of".

Claim 1 of auxiliary requests 6 and 7 differ from those of the main request and the first auxiliary request respectively in that component b. is defined as follows:

"...b. at least one substantially crosslinked alkali-swellable acrylate copolymer rheology modifier polymerized derived from
(i) 20.35% to 30.65% by weight of acrylic acid or methacrylic acid, or combinations thereof,
(ii) 30.65% to 45.35% by weight of ethylacrylate, or methylacrylate, or combinations thereof, and
(iii) from 0.010.03% to 53% by weight of at least one of polyalkenyl ethers of sucrose or polyalcohols; or trimethylolpropane tri(meth)acrylate, glycidyl methacrylate, N-methylolacrylamide, or combinations thereof."

Claim 1 of auxiliary requests 8 and 9 differ from those of the auxiliary requests 6 and 7 respectively in that the wording "derived from" in feature b. has been replaced by "polymerized from the three components (i), (ii) and (iii)".

Claim 1 of auxiliary requests 10 and 11 differ from those of the auxiliary requests 6 and 7 respectively in that the wording "derived from" in feature b. has been replaced by "polymerized from a monomer composition consisting of".

XI. The relevant arguments of the appellant and opponent 01 may be summarised as follows:

Main request - Inventive step of claim 1

D3 was a suitable starting point for the skilled person. Example 5 of D3 differed from claim 1 in that in the former, the process of back-acidification is not carried out. Even if the alleged technical effects of providing suspension stability and improving rheological properties in acidic pH had been sufficiently demonstrated, it would not have rendered the claims inventive, since said effects were to be expected in view of the teaching of D1. Accordingly,
the skilled person knew from D3 that the cross-linked copolymers thereof possessed low viscosity in the acid form, and that neutralisation was required to produce the thickening effect. D1 taught the skilled person that if a back-acid activation was performed, the thickener in question displayed higher viscosity at low pH than the equivalent mixture when acidified without first being neutralised. Thus starting at the disclosure of D3 and faced with the technical problem of providing the aforementioned effects, the skilled person, with reasonable expectation of success, would have attempted the back-acid activation procedure described in D1 on the products of D3 in order to solve the problem, and thereby would have arrived at the subject-matter of claim 1 without exercising inventive skill.

Auxiliary requests - Inventive step

The arguments submitted in respect of inventive step for claim 1 of the main request applied equally to the corresponding claims of the auxiliary requests.

Opponent 03 did not intervene in the proceedings.

XII. The relevant arguments of the respondent may be summarised as follows:

Main request - Inventive step of claim 1

D3 was a more appropriate starting point than D1 for the assessment of inventive step. D1 did not concern the long term stabilisation of insoluble matter in suspension, and drew a distinction between so-called HASE polymers according to the invention, and ASE polymer mixtures L-O (table II) which comprised a
copolymer of methacrylic acid and ethyl acrylate, lacked an associative monomer and were merely comparative. D3 on the other hand addressed the problem of long-term suspension stability in an aqueous surfactant-containing composition, the same technical problem as that underlying the contested patent (D3, page 5, lines 80-100). Furthermore D3 (example 5) disclosed the same copolymer of methacrylic acid and ethyl acrylate used according to mixtures L-0 of D1, but additionally including a cross-linker. Claim 1 differed from D3 by the back-acidification step. The effect of the difference was demonstrated by the experimental data report filed with the letter of 22 October 2013. Accordingly, the example "EX Inv" was equivalent in composition to example 5 of D3, comprising 60% ethyl acrylate, 40% methacrylate, and a cross-linker. According to figures 1-3 of said report, the aqueous surfactant-containing composition tested displayed increased viscosity and yield value, and improved suspension stability on progressive lowered of the pH by acidification. The objective technical problem was the provision of an aqueous surfactant composition which provides for improved rheological properties and suspension stability, and is effective in the acidic pH range. The solution provided by claim 1 was not obvious in view of D3, which teaches that in order to act as thickeners, the copolymers as defined must be in the carboxylate or neutralised (sodium salt) form. This teaching was supported by D6 (page 235, part a) which specified that copolymers of acrylic acid all required neutralisation of their carboxylic functionality with base to facilitate effective elongation of the polymer chains for the purpose of thickening. Furthermore, the solution was not obvious in view of D1, the aim of which was to increase the viscosity of aqueous compositions, and not the long-
term suspension stability thereof, which was unrelated. Even if the skilled person would have taken D1 into account, he would have started from the core teaching thereof relating to the HASE polymers, and not the data provided for the ASE polymers L-0. D1 furthermore taught that upon re-acidification, most aqueous thickener solutions will coagulate and revert to low viscosity suspensions (column 3, lines 25-27), thereby teaching against the acidification of ASE copolymers and away from the invention. Consequently, claim 1 involved an inventive step.

Auxiliary requests - Inventive step

The arguments submitted in respect of inventive step for claim 1 of the main request applied equally to the corresponding claims of the auxiliary requests.

XIII. The appellant requests that the decision under appeal be set aside and the patent be revoked.

XIV. The respondent requests that the patent be maintained according to the set of claims filed as main request or alternatively according to the sets of claims of auxiliary requests 1 to 11, all filed with the letter of 7 June 2015.

Reasons for the Decision

Main request - Inventive step of claim 1

1. According to the specification, the claimed invention relates to stable aqueous compositions comprising a surfactant, a cross-linked alkali-swellable acrylate
copolymer rheology modifier and water in which a substantially insoluble compound can be stabilised or suspended (paragraph [0011]).

Closest prior art

1.1 While in written proceedings the respondent had argued that D1 represents the closest prior art, during oral proceedings before the Board the view was taken that D3 represented the most appropriate starting point for the skilled person. Since the appellant agreed and the Board see no reason to differ, D3 is chosen as closest prior art.

1.2 D3 is directed to aqueous emulsion copolymers and thickening procedures using them, and discloses a composition comprising 1.5% sodium lauryl sulfate (Emulsifier B, a surfactant), a copolymer prepared from 40% methacrylic acid and 60% ethyl acrylate (components (i) and (ii) of claim 1) and 0.2% of a polyallyl ether of sucrose as cross-linker (component (iii) of claim 1) (D3, example 5, and table III, first entry).

1.3 The parties agree that this composition differs from that of claim 1 of the main request in that it has not been back-acidified (i.e. neutralised with an alkaline material, and re-acidified with an acidic material) according to the instructions laid out in said claim.

Problem solved

2. According to the patent specification, the general object of the invention is to provide aqueous surfactant compositions which adequately stabilise substantially insoluble compounds or materials (paragraphs [0002] and [0009]).
2.1 In order to formulate the objective technical problem effectively solved by the claimed subject-matter, it must be determined whether the distinguishing features of the claim provide the alleged technical effects or advantages. Thus it must be investigated whether the alleged effects of increased viscosity, yield value and improved suspension stability are supported by the evidence on file.

2.2 As to the evidence provided in the patent specification, the "Comparative Stability Testing" (paragraphs [0100] to [0115]) is not suitable for providing evidence of an effect by virtue of the non-identification of the polymer W, which is denoted merely "Acrylates crosspolymer". Indeed, it cannot be excluded that said polymer no longer falls within the scope of those recited in claim 1 of the main request, the definition of which is severely limited compared to that of claim 1 as originally filed.

2.3 The respondent refers to the Supplementary Experimental Data report filed with the letter of 22 October 2013 as evidence of the alleged effects. The Board considers it reasonable to view test "EX Inv" of said report as equivalent in composition to the copolymer dispersions of D3, example 5, since the monomers are identical and are employed in the same ratio, comprising 60% ethyl acrylate, 40% methacrylate, and a cross-linker. The other compositions tested according to said report are not relevant since they include an associative monomer, which is implicitly excluded from the scope of the copolymers of claim 1 by virtue of the closed definition thereof ("polymerized from"). Figures 1 and 2 of the report demonstrate increasing viscosity and yield value when the pH is progressively lowered by
acidification. Furthermore, the viscosity and yield values remain relatively high after 13 weeks at 45°C. Figure 3 demonstrates that as the pH was lowered to pH 5 and 4 by back acid addition with citric acid, the composition met the suspension stability criteria at 13 weeks.

2.4 Although according to the specific test described above, the viscosity and yield value of the composition in question increased as the pH was lowered, the Board does not consider it plausible that the effect demonstrated for this specific copolymer is achievable across the entire scope of claim 1, which covers copolymers prepared from other monomers, and in widely different weight percentage ratios. Not only is claim 1 very broad compared to the test example, but it is also explicitly stated in the patent specification in relation to the effect of the back-acid technique that "..if these compositions are raised to a neutral or even alkaline pH and then subsequently reduced in pH, the viscosity and yield value generally remain unchanged or often actually increase" (emphasis added) (paragraph [0070]). Thus, although some embodiments, such as that tested, demonstrate the increase, others will not. Nevertheless, in view of the test data discussed above, as well as the results presented in D1 in respect of the effect on viscosity of back-acidification of the mixtures L-0 (D1, table 3), the Board accepts as plausible and thus achievable across the scope of claim 1 that the viscosity and yield value are not negatively affected upon lowering of the pH by acidification.

2.5 The same reasoning applies to the plausibility of the effect of improved suspension stability. Additionally, in this regard, although figure 3 of the test report
mentions that the copolymer thereof failed the suspension stability test at the initial "base neutralised" pH value of 6.5, it is difficult to accept this as evidence that the polymer compositions according to D3 will not display sufficient suspension stability, since although D3 teaches the necessity of neutralisation, it also explicitly teaches that the degree of neutralisation of the carboxylic acid groups in the copolymer must be adjusted to produce the desired thickening effect and may be complete, but may also be as low as 50% (D3, page 4, lines 71-86). Since a link has been established between suspension stability and rheological properties (vide infra, point 3.4), it cannot be excluded that the test at pH 6.5 is not optimised in terms of thickening, and thus suspension stability. Thus the Board accepts as plausible that the effect of sufficient suspension stability is achievable across the scope of claim 1.

2.6 It follows that the objective technical problem underlying claim 1 is the provision of an aqueous surfactant composition effective in the acidic pH range and providing a sufficiently stable suspension of insoluble material while not adversely affecting the rheological properties thereof.

Obviousness

3. The aforementioned problem has been solved by adding an acidic material as defined in claim 1 to the (partially) neutralised copolymer dispersions of D3.

3.1 D1 discloses a method for thickening low pH aqueous surfactant-containing compositions by using a back-acidification process (termed "activation" therein) whereby it is taught that in the presence of a
surfactant, if the solution in question is first neutralised and subsequently acidified, then coagulation does not occur and the acidified composition is thickened, and thus "activated", i.e. it retains a significantly higher viscosity than that of the equivalent mixture which is acidified without first being neutralised (D1, column 3, lines 19-40). Example II of D1 discloses mixtures A-K, which comprise an associative monomer, while others (specifically mixtures L, M, N and O) comprise copolymers of methacrylic acid and ethyl acrylate and do not include an associative monomer.

3.2 While the appellant argues that the solution to the aforementioned problem is to be found in the teaching of D1, the respondent disagrees, arguing that in order to solve the technical problem posed, the skilled person would not have turned to the teaching of D1 for the solution. D1 did not address the problem of long term suspension stability, but rather only that of viscosity, which was unrelated. Furthermore, even if the skilled person were to consult D1, he would have turned to the core teaching thereof, which concerned HASE polymers, and not the comparative data presented in table II relating to the ASE polymer mixtures L-O which correspond to the copolymer mixtures of the contested patent, but without a cross-linker.

3.3 D3 itself however teaches the following in respect of the cross-linked copolymers thereof (page 5, lines 88-100):

"These lightly cross-linked materials are unique in the water-soluble polymer field in regard to their suspending action for pigments, abrasives, ceramics, etc. Unlike conventional polyacrylate or natural gum
thickeners, it is possible with the lightly cross-linked emulsion copolymers of the present invention to thicken suspensions of pigments to relatively low viscosities (500 to 100 cps., Brookfield 12 R.P.M., 25°C.) and yet obtain stability in the suspension as measured by lack of separation" (emphasis added)

3.4 Thus, while conventional polyacrylate copolymers require higher viscosities to stabilise suspensions, the cross-linked copolymers according to D3 possess suspension stability at low viscosities. This statement thus acknowledges that for any given copolymer, there is a (albeit unquantified) link between the viscosity thereof and the suspension stability. This position appears to be supported by the common general knowledge of the skilled person represented by D6, which states that cross-linking creates a yield value that non-crosslinked copolymers lack, and that consequently, their solutions are able to aid in stabilizing the dispersed phase on oil-in-water emulsions, pigments, particle... and any other component that requires suspension (D6, page 235, last paragraph). Thus the skilled person would understand from D3 that in order for the crosslinked copolymers to stabilise suspensions, a certain level of thickening (viscosity and/or yield value) is required. It follows that viscosity and suspension stability are not unrelated as alleged by the respondent.

3.5 The viscosity data presented for the ASE copolymer mixtures L-O of D1 (tables II and III) demonstrates that said mixtures also respond to back-acid activation, since on sequential lowering of the pH by addition of citric acid, their viscosities either remain similar, or increase. In view of this teaching, whether said mixtures were intended to be according to
the invention of D1 or, as argued by the respondent, merely for comparative purposes, is irrelevant.

3.6 Furthermore, the fact that the copolymers of D3 are cross-linked would not discourage the skilled person from applying the back-acidification process taught by D1 to them, since D1 indicates that it is the free acid groups, present in the cross-linked copolymers, which are critical for said process (D3, column 4, lines 23-28).

3.7 Thus there is no apparent reason why the skilled person, with reasonable expectation of success, would not consider applying the back-acidification process of D1 to the copolymer dispersions of D3 in order to solve the aforementioned objective technical problem.

3.8 It follows from the above that claim 1 of the main request does not meet the requirements of Article 56 EPC.

Auxiliary requests - inventive step of claim 1

4. As is apparent from the rationale provided in the letter of the respondent dated 7 June 2015, the purpose of the amendments to claim 1 of each of the respective auxiliary requests was not to establish inventive step, should the main request fail in this respect, but rather to overcome further grounds (in particular Article 123(2) and (3) EPC) raised by the opponent in appeal proceedings. This understanding was confirmed during oral proceedings before the Board whereby the respondent agreed with the statement of the chairman that the same arguments in respect of inventive step of claim 1 of the main request applied to claim 1 of each of the respective auxiliary requests. In this context,
it is not necessary for the board to comment further on
the merits of the individual requests in respect of
inventive step.

4.1 It follows that auxiliary requests 1-11 fail to meet
the requirements of Article 56 EPC for the same reasons
as those provided for the main request.

Conclusion

5. Since none of the requests on file meet the
requirements of Article 56 EPC, the patent is to be
revoked and the Board does not need to decide on any
other issue.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.

2. The patent is revoked.
The Registrar: S. Fabiani
The Chairman: J. Riolo

Decision electronically authenticated