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**Datasheet for the decision
of 24 January 2018**

Case Number: T 1176/14 - 3.3.03

Application Number: 04740047.8

Publication Number: 1639021

IPC: C08F4/00, C08F2/22

Language of the proceedings: EN

Title of invention:

POLYMERIZATION PROCESS FOR PREPARING (CO) POLYMERS

Patent Proprietor:

Akzo Nobel Chemicals International B.V.

Opponent:

SOLVAY (SOCIETE ANONYME)

Relevant legal provisions:

EPC Art. 56

Keyword:

Inventive step - (yes)



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Case Number: T 1176/14 - 3.3.03

D E C I S I O N
of Technical Board of Appeal 3.3.03
of 24 January 2018

Appellant: Akzo Nobel Chemicals International B.V.
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Decision under appeal: **Decision of the Opposition Division of the
European Patent Office posted on 25 March 2014
revoking European patent No. 1639021 pursuant to
Article 101(3) (b) EPC.**

Composition of the Board:

Chairman D. Semino
Members: M. C. Gordon
C. Brandt

Summary of Facts and Submissions

- I. The appeal of the patent proprietor lies from the decision of the opposition division posted on 25 March 2014 revoking European patent number 1 639 021.
- II. The patent was granted with a set of 14 claims, whereby claim 1 read as follows:

"Polymerization process for preparing a (co)polymer wherein one or more organic peroxides selected from the group consisting of diacyl peroxides, peroxyesters, peroxydicarbonates, and mixtures thereof are used in conjunction with 0.3-45 wt%, based on the weight of organic peroxide, of one or more controlling agents selected from the group consisting of organic hydroperoxides, ethylenically unsaturated organic compounds that preferably cannot homopolymerize, compounds with labile carbon-hydrogen bonds, oximes, and mixtures thereof, with the proviso that the solubility of the peroxydicarbonate(s) in water at 0°C is at least 5 ppm, preferably the solubility of all organic peroxides in water at 0°C is at least 5 ppm, and wherein the process is an aqueous dispersion polymerization process wherein at least part of the one or more organic peroxides used as initiator is dosed to the reaction mixture at the polymerization temperature."

Claims 2-11 were dependent on claim 1. Claim 12 was directed to a (co)polymer obtainable with the process of one of claims 1-11, and claims 13 and 14 were directed to a formulation suitable for use in the process.

III. A notice of opposition was filed in which revocation of the patent on the grounds of Article 100(a) EPC (lack of novelty, lack of inventive step), Article 100(b) EPC and Article 100(c) EPC was requested.

The following documents, *inter alia* were cited in support of the opposition:

D4: US-A-6 399 728

D7: US-A-2002/0123591

D10: US-A-3 778 422.

IV. The decision of the opposition division was based on three amended sets of claims forming a main request and first and second auxiliary requests.

Claim 1 of the first auxiliary request read as follows, deletions compared to claim 1 of the patent as granted being indicated in ~~strikethrough~~ and additions in **bold**:

"Polymerization process for preparing a (co)polymer wherein one or more organic peroxides selected from the group consisting of diacyl peroxides, ~~peroxyesters, peroxydicarbonates, and mixtures thereof~~ are used in conjunction with 0.3-45 wt%, based on the weight of organic peroxide, of one or more controlling agents selected from the group consisting of organic hydroperoxides, **and dibutyl maleate** ~~ethylenically unsaturated organic compounds that preferably cannot homopolymerize, compounds with labile carbon hydrogen bonds, oximes, and mixtures thereof, with the proviso that the solubility of the peroxydicarbonate(s) in water at 0°C is at least 5 ppm, preferably the solubility of all organic peroxides in water at 0°C is at least 5 ppm,~~ and wherein the process is an aqueous dispersion polymerization process wherein at least part

of the one or more organic peroxides used as initiator is dosed to the reaction mixture at the polymerization temperature."

All requests retained a claim directed to the (co)polymer obtainable by the process of any of the process claims.

- V. According to the decision the main request did not meet the requirements of clarity and sufficiency of disclosure. The details of these objections are not relevant for the present decision.

As far as the first auxiliary request was concerned, whilst the clarity and sufficiency objections had been addressed, the claims directed to a copolymer did not meet the requirements of Article 54 EPC. With respect to inventive step for the process of claim 1, it was held that D7 represented the closest prior art, the distinguishing feature being the use of the specific controlling agent. The technical effect thereof was a reduction in fish eye content. D10 disclosed that when using "fast initiators" the generation of heat exceeded the system's cooling capabilities, resulting in a "heat kick". This could be minimised by incorporation of controlling agents such as an alkyl hydroperoxide in the system, leading to a more stable polymerisation temperature and thus to polymers of more uniform molecular weight. The decision then stated - without reference to any supporting documents - that it was known that polymerisation temperature strongly influenced the molecular weight of the produced polymer and that it was also known that areas of higher molecular weight within a polymer could lead to fish eyes. Thus, it was concluded, D10 taught how in a process according to D7 fish eyes could be reduced. An

indication was also provided by D4 which taught that use of e.g. dibutyl maleate stabilised a peroxy dicarbonate against decomposition, which according to the patent in suit was precisely what the controlling agent was intended to achieve. On that basis process claim 1 was found to lack an inventive step.

The same conclusion applied to the second auxiliary request.

Consequently the patent was revoked.

VI. The patent proprietor lodged an appeal against the decision.

Together with the statement of grounds of appeal four sets of claims forming a main and three auxiliary requests were submitted.

The main request corresponded to the first auxiliary request underlying the decision, amended by deletion of the claim directed to the (co)polymer obtainable by the process.

The details of the further requests are not of relevance for this decision.

A further document:

D13: Encyclopedia of PVC (L.E. Nass, Ed) 1976, pages 76-85

was submitted.

VII. Together with the rejoinder the respondent submitted a document:

D14: Encyclopedia of Polymer Science and Engineering, Volume 17, 1989, John Wiley & Sons, pages 295-319 and 328-329.

VIII. The Board issued a summons to attend oral proceedings and a communication. In the preliminary view of the Board the principal matter to be addressed was the occurrence of fish eyes and in particular the question of whether, as held by the decision, "heat kicks" during polymerisation were known to be responsible for this phenomenon. It appeared that only the patent in suit postulated such a link; the documents cited, including the newly cited D13 and D14 were however silent in respect thereof.

IX. The respondent made a further written submission and provided a further document:

D15: Lukas, R. Makromol. Chem., Macromol. Symp 29, 21-40 (1989).

X. Oral proceedings took place before the Board on 24 January 2018.

XI. The arguments of the appellant can be summarised as follows:

Fish eyes were spots of polymer with higher molecular weight than the surrounding polymer but did not arise as a result of heat kicks. No document provided a link between heat kicks and fish eye formation. D10 addressed the question of uniformity of molecular weight and structure but did not concern fish eye formation. Nor did any other of the cited documents provide a link between the types of "non-uniformity"

considered in D10 and fish eye formation. There were many possible types and sources of "non-uniformity" in polymers and D10 did not provide a complete or detailed discussion of these. It was known that the molecular weight of PVC depended on the polymerisation temperature, higher temperature leading to lower molecular weight (D13, D14). Thus reduction of heat kicks would not lead to reduction of fish eye formation. Support for this position was provided by D15 which showed that fish eyes were associated with regions of equal or higher molecular weight and postulated various possible sources thereof, e.g. non-uniform distribution of initiator resulting in generation of "difficultly processible" regions or particles, but did not however refer to heat kicks. D13 associated the occurrence of fish eyes with impurities in the reactor (incomplete removal of previous charges of polymer) leading to gel like particles. Thus the prior art provided no link between heat kicks and fish eye formation. On that basis there was a missing link in the reasoning of the opposition division and the presence of an inventive step should be acknowledged.

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

The cause of fish eye formation was not completely understood. According to the patent these arose as a result of inhomogenities, i.e. regions of differing molecular weight or melting point to the surrounding regions. D10 taught that heat kicks during polymerisation, and hence the resulting inhomogenities in the obtained polymers, could be avoided by use of controlling agents. D13 confirmed the problem of the occurrence of heat kicks in production of PVC. D14 taught various possible sources of fish eyes - use of

an incorrect colloid, poor agitation, doubly polymerised particles arising from contamination from previous polymer charges in the reactor, leading to particles of differing - in particular higher - molecular weight or porosity than the surrounding polymer. Alternatively D15 suggested that fish eyes were the result of regions of higher molecular weight and that overheating resulted in glassy structures. From this it could be deduced that overheating would be expected to give rise to fish eyes, which was a further aspect relating to the uniformity of the polymer to which D10 related. All of these teachings expressed - in different ways - the need to ensure a uniform polymerisation process in order to avoid fish eye formation, and D10 explained how to accomplish this, i.e. by use of a controlling agent. The reasoning of the opposition division was therefore correct and claim 1 lacked an inventive step.

XIII. The appellant requested that the decision under appeal be set aside and that the patent be maintained in amended form on the basis of one of the sets of claims according to the main request or first to third auxiliary requests all filed with the statement setting out the grounds of appeal.

XIV. The respondent requested that the appeal be dismissed.

Reasons for the Decision

1. Admittance of the documents cited during the appeal proceedings.

No objections were raised in respect of the admittance of any of documents D13 (appellant) or D14 and D15

(respondent).

Indeed both parties relied on all these documents - regardless of provenance - in their argumentation.

Accordingly the Board can identify no reason to exclude any of D13-D15 from the procedure.

2. Main request - inventive step

2.1 Closest state of the art, distinguishing feature

It is a matter of consensus between the parties that the closest prior art is represented by D7 which discloses, in particular in Table II, a process exhibiting all the features of operative claim 1 with the exception of the presence of the component identified as the controlling agent, which therefore constitutes the distinguishing feature. D7 also explicitly addresses the problem of fish eye formation and identifies as a means to avoid or reduce this, use of a "proper" peroxide (paragraph [0005]) by which is meant, *inter alia*, a diacyl peroxide as required by operative claim 1 (D7, paragraph [0011] and examples).

2.2 Technical effect

It is also a matter of consensus between the parties that the effect of this difference, as demonstrated by the examples of the patent, is to increase the time until peak exotherm and also to reduce (further) the number of "fish eyes" i.e. visible defects in the obtained polymer (PVC in the examples of the patent and of D7).

2.3 Objective technical problem

The provision of a process for the preparation of a polymer with - further - reduced fish eye content thus represents the objective technical problem to be solved with respect to D7.

2.4 Obviousness

According to D7 fish eye formation is linked to the use of particular peroxides (see above). According to paragraph [0005] of D7 by use of the "proper" peroxide and dosing conditions it is possible to obtain a polymerisation reaction with virtually constant heat of polymerisation allowing efficient use of the reactor and peroxide, high yields, low residual peroxide levels, low reactor fouling and low fish eye content. D7 does not offer any analysis of the mechanism giving rise to the fish eyes beyond the link to the peroxide or any explanation why the disclosed process results in products with a low content of fish eyes.

D10 addresses the avoidance of heat kicks during polymerisation initiated by organic peroxy dicarbonates, i.e. different initiators to those common to the operative claims and D7, which heat kicks are stated to arise as a result of auto-acceleration of the polymerisation (column 1, lines 18-44). It is taught that because molecular weight, branching, heat stability and other properties of vinyl halide polymers are affected by the polymerisation temperature, a relatively constant temperature, and hence avoidance of heat-kicks, is necessary to obtain a uniform product.

This is achieved according to D10 by inclusion of an alkyl hydroperoxide in the reaction system (column 1,

line 69 to column 2, line 3) which results in the reaction temperature being restricted to a narrow range leading to a polymer of more uniform molecular weight and structure (column 2, lines 10-14). D10 does not relate to polymerisation initiated by diacyl peroxides as required by operative claim 1 and contains no mention of fish eyes. It has not been shown by the respondent that any aspect of D10 provides a teaching - even indirect - with respect to fish eye formation.

D4 also relates to the stabilisation of dialkyl peroxydicarbonates in peroxide-initiated polymerisation of vinyl monomers (D4 column 1, lines 3-25. This is achieved according to D4 by inclusion of non-polymerisable diesters of unsaturated dicarboxylic acids (column 1, lines 20-23; claim 1). According to column 4, lines 1-10 the diester suppresses self-accelerating decomposition of the peroxydicarbonate during manufacture and enhances the thermal stability thereof during subsequent storage and handling. D4 however contains no consideration of the influence of the presence of the diester on the course of polymerisation or on the characteristics of the resulting polymers and no discussion of fish eye formation.

D13 relates to the formation of PVC and teaches that high temperature results in low molecular weight polymer (page 79, second paragraph). Heat kicks are discussed at page 78 in the fourth paragraph and linked to auto acceleration of peroxide decomposition, consistently with D10. D13 states that this happens at the end of the reaction. At page 79, second paragraph it is reported that higher polymerisation temperature results in lower molecular weight polymer, meaning that for efficient polymerisation adequate cooling is

necessary. The means of controlling the heat in the reactor are discussed on page 78, 2nd and 3rd paragraphs. At page 84, third complete paragraph the foregoing is confirmed, as it is taught that to attain low molecular weight polymers high temperatures have to be used. At page 85, second complete paragraph, the formation of fish eyes is discussed. These are described as "small particles that do not readily fuse, thus remaining as discrete gel-like particles". D13 states that whilst fish eyes can occur at any stage of the polymerisation, cleanliness of the reactor is important since polymer particles adhering to the reactor wall serve as nuclei for fish eyes. The following paragraph states - consistently with the teachings of D7 - that some initiators have a tendency to produce more fish eyes than others, and that the suspending system has an influence. In the following paragraphs, the discussion returns to control of the resin molecular weight and this is identified as of prime importance in determining the end use of the resin. It is again confirmed that the molecular weight is controlled by the temperature (and the level of chain transfer agent). In the following paragraph heat stability is discussed and it is stated that poor heat stability can result from overheating of the resin, heat kicks or a drying temperature that is too high.

Thus according to the teaching of D13 fish eyes are primarily caused by impurities in the reactor. Although temperature control and heat kicks are discussed in D13 and the consequences for the properties of the polymer (lower molecular weight) are discussed, no link is made between these process aspects and fish eye formation.

D14 teaches that an increase in temperature leads to a reduction in molecular weight (page 301, paragraph

above Table 2). In this statement, D14 is consistent with D13. D14 further teaches in the same paragraph that the porosity of the polymer is also decreased by higher temperature. Fish eye formation is discussed in D14 at page 302 in the second complete paragraph. This is stated to be caused by "various polymerisation" parameters such as incorrect choice of protective colloids or poor agitation. However, and again consistently with D13, the most common cause is identified as being impurities, i.e. improper cleaning of the reactor leading to doubly polymerised particles. D14 contains no recognition - even implicit - of a link between heat kicks and fish eye formation.

D15 addresses the phenomenon of fish eyes in PVC and seeks to provide a means to identify the presence of precursor particles of fish eyes ("PFE" in the terminology of D15). It is considered that these are caused by the presence of "difficultly processible particles" (page 22, lower section). It is stated therein that the problem of the occurrence of these particles has not been solved, but it had been assumed that the source was the presence of a high content of strongly branched or crosslinked macromolecules of PVC. In D15 the process was manipulated to ensure an increased content of PFE particles. According to page 23, second and third full paragraphs these PFE particles were identified and extracted. It was found that these precursor particles were heterogenous, having a non-porous glassy structure. According to page 26, first partial paragraph, the PFE particles were found to contain neither branched, nor crosslinked macromolecules, contradicting previous understanding (see discussion of page 22 of D15 above). According to page 26, section entitled "Formation of PFE particles" it was found that these had an elevated content of

conjugated double bonds, resulting in reduced thermal stability. In the paragraph bridging pages 26 and 27 overheating of the polymerisation reaction is dismissed as a source of PFE formation. It is then postulated that non-homogeneous distribution of initiator might be a cause of the PFE particles (page 27, first partial paragraph, last 5 lines). This hypothesis was confirmed by means of the experiments reported in the following section on page 28. This is also one of the conclusions of the whole paper (page 31).

From the various secondary documents it follows that two primary sources of fish eye formation have been identified:

- impurities in the reactor resulting from residual charges of previously formed polymers
- inadequate distribution of initiator between the monomer.

However conclusions as to other causes of fish eyes are indeterminate and, to an extent contradictory, or appear to lead to a conclusion that fish eyes would not arise. In particular the teaching with respect to heat kicks is that these would be expected to result in areas of lower molecular weight but there is no recognition of any link to fish eye formation.

Although it might be hypothesised that in particular the question of homogeneous distribution of initiator might have an influence on the heat management of the system, this does not emerge from D15 or any other document. Similarly D10 does not provide any insight as to the mechanism by which the addition of the alkyl hydroperoxide reduces the instances of "heat kicks". Nor does the observation of D10 that the alkyl

hydroperoxide leads to a more constant temperature resulting in a more uniform product provide any link to fish eyes, even taking into account the discussion of D15 relating to the need for homogeneous distribution of initiator, since according to D15 the very effect which D10 seeks to avoid - heat kicks - is dismissed as being a potential source of fish eyes.

It therefore does not emerge from the prior art that there is any link between the temperature management during the polymerisation reaction (elimination of heat kicks) and the formation of fish eyes. Nor do any of the documents provide any indication that formation of fish eyes could be suppressed by some other mechanism by including in the polymerisation system the specified initiator in combination with an organic hydroperoxide or dibutyl maleate.

Hence there was no incentive for the skilled person seeking to reduce the incidence of fish eyes arising in the process of D7 to modify the process thereof by incorporating a member of either of the classes of compounds known from D10 and D4 to influence the stability of peroxy initiators and designated in the patent in suit as "controlling agent".

The Board therefore comes to the conclusion that the subject-matter of claim 1 involves an inventive step over the available prior art.

2.5 The main request therefore meets the requirements of Article 56 EPC.

Order

For these reasons it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the opposition division with the order to maintain the patent in amended form on the basis of claims 1 to 10 according to the main request filed with the statement of grounds of appeal and after any necessary consequential amendment of the description.

The Registrar:

The Chairman:



B. ter Heijden

D. Semino

Decision electronically authenticated