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File No.: T 0104/90 - 3.3.3
Application No.: 83 108 780.4
Publication No.: 0 103 261
Classification: C08G 75/02
Title of invention: Continuous extraction of polyphenylene sulfide

D E C I S I O N
of 19 July 1993

Applicant: PHILLIPS PETROLEUM COMPANY
Proprietor of the patent:
Opponent:

Headword:

EPC: Art. 56

Keyword: "Inventive step (yes)"

Headnote
Catchwords



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Boards of Appeal

Chambres de recours

Case Number: T 0104/90 - 3.3.3

D E C I S I O N
of the Technical Board of Appeal 3.3.3
of 19 July 1993

Appellant: PHILLIPS PETROLEUM COMPANY
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Decision under appeal: Decision of the Examining Division of the European
Patent Office dated 13 September 1989 refusing
European patent application No. 83 108 780.4
pursuant to Article 97(1) EPC.

Composition of the Board:

Chairman: F. Antony
Members: R.A. Lunzer
J.A. Stephens-Ofner

Summary of Facts and Submissions

I. European patent application No. 83 108 780.4, publication No. 0 103 261, was filed on 6 September 1983, having a priority date of 10 September 1982 derived from US application No. 416 756.

II. Before the Examining Division there were in all seven claims, the single independent Claim 1 being in the following form:

"A method for extracting water soluble impurities from an aqueous slurry of arylene sulfide polymer containing water soluble impurities, characterized by

- (1) continuously mixing said polymer containing impurities with water to produce a slurry containing from 3 to 30 percent by weight poly(arylene sulfide),
- (2) continuously heating a flow of said slurry by passing it through indirect heat exchange contact with hot heat exchange medium to raise the temperature of said slurry to a range from 182 to 216°C, and
- (3) continuously separating said polymer from said slurry."

By its decision given on 13 September 1989, the Examining Division refused the application, holding that although the alleged invention the subject of Claim 1 then under consideration was novel, it lacked any inventive step having regard to the closest prior art:

- (1) US-A-4 025 496.

The Examining Division found that the Appellant had not produced any satisfactory evidence that an existing prejudice against continuous operation had been overcome. It did not find the statement contained in a declaration by Lacey E. Scoggins convincing (see point III below). Furthermore, it was not satisfied that the results in Table I, page 5, of the application sufficiently demonstrated that the continuous process in accordance with the alleged invention was a significant improvement on the batch process of the prior art.

III. An appeal against that decision was lodged on 21 November 1989, the appeal fee was paid on the same day, and the Grounds of Appeal were filed on 23 January 1990. In the Statement of Grounds of Appeal, the Appellant argued that the alleged invention had the advantages of saving both processing time and energy consumption, and had overcome a prejudice in the industry against the practical feasibility, for the purpose concerned here, of continuous processes, as confirmed by the affidavit of Lacey E. Scoggins filed in the course of examination on 17 August 1988. PPS (polyphenyl sulphide) when produced by the existing technical methods is necessarily associated with an approximately equal amount of common salt. Although the first stages of salt removal are relatively easy, because the salt is caked onto the PPS particles, the removal of the remaining salt is more difficult because the salt is occluded in the PPS particles. To overcome that problem the practice used hitherto had involved multiple stages of batch washing with agitation over an extended period of time, followed by filtration. It was hitherto believed that extended contact time, coupled with intensive stirring of the slurry, were both necessary for adequate salt removal.

IV. Communications from the Board dated respectively 10 August 1992 and 22 October 1992, drew attention to the fact that although it appeared from parts of the description, notably Example II and the figures for ash content given with respect to the feed material, that the alleged invention was concerned with the final stages of salt removal, rather than the removal of the gross amounts initially associated with the PPS, the claims were not restricted to that later step, but seemed to cover salt removal in general. The Board also drew the Appellant's attention to the necessity to incorporate the features of Claims 2 and 3 of the application as filed into Claim 1 for the sake of consistency with the case argued on appeal, and supported by the declaration of Mr. Scoggins.

V. With its letters of 13 and 15 July 1993, the Appellant filed a revised set of claims, Claim 1 being in the following form:

"A method for extracting water soluble impurities from an arylene sulfide polymer, which has undergone prewashing warm water operations to remove a substantial portion of said impurities,

characterized by

- (1) continuously mixing said prewashed polymer containing remaining impurities with water to produce a slurry containing from 3 to 30 percent by weight poly(arylene sulfide),
- (2) continuously heating a flow of said slurry by passing it through indirect heat exchange contact with hot heat exchange medium to raise the temperature of said slurry to a range from 182 to 216°C,

- (3) extracting a substantial portion of said remaining impurities during a residence time at said temperature thereby deashing said polymer, and
- (4) continuously separating said polymer from said slurry,
- (5) said slurry being cooled by indirect contact with the heat exchange medium after having been heated to said temperature of 182 to 216°C before separating of polymer from water,
- (6) a closed cycle of heat exchange medium being employed to at least partially heat and to at least partially cool said slurry."

VI. The Appellant requested that the decision under appeal be set aside, and a patent granted on the basis of Claim 1 as set out above, with Claims 2 to 5 dependent thereon, but otherwise corresponding exactly with Claims 4 to 7 of the application as filed.

Reasons for the Decision

1. The appeal is admissible.
2. *Admissibility of amendments*
 - 2.1 The first important difference between Claim 1 as amended, and in the application as filed, is that the preamble to the amended claim has been limited to a feed polymer which has undergone "prewashing warm water operations to remove a substantial portion of said

impurities", and the first step of the characterising part of the claim involves mixing said "prewashed" polymer.

Example I, which describes the process of the alleged invention in connection with Figure 1, refers to the use as feed material of PPS that had already undergone several warm water washing operations being mixed as the first step of the process in stirred tank (1).

Example II refers to material which had been deashed by the process of Example I, and identifies the said feed as having an ash content of 1.6 to 1.8%. In the light of these disclosures, the Board is satisfied that the application as filed disclosed the alleged invention as being applicable, if not exclusively applicable, to the final stages of the removal of salt from PPS polymers, from which it follows that the positive inclusion of this prewashing step into Claim 1 is admissible for the purposes of Article 123(2) EPC.

2.2 Step (3) specifies the extraction of a substantial portion of the remaining impurities inherent to the entire disclosure, while step (4) relates to the continuous separation, which was step (3) of the claim considered by the Examining Division.

2.3 The other amendments to Claim 1 essentially involve incorporating verbatim the features of original Claims 2 and 3, so as to make it clear that recuperative heat exchange in a closed cycle is now an essential feature of the alleged invention. The introduction of these features into Claim 1 is clearly admissible for the purposes of Article 123(2) EPC.

3. *Novelty*

Having reviewed the cited documents, the Board is satisfied that none of them discloses a process having all the features of Claim 1 as now formulated.

4. *The closest prior art*

4.1 The Board agrees with the Examining Division in starting from document (1) as the closest prior art. That document discloses a process for producing and purifying PPS in which the reaction mixture is washed with sufficient water to extract most of the salt, and filtered. According to the detailed description, the washing and filtration steps are repeated one to four times, and although washing at ambient temperatures is possible, it is said to be preferable for at least one of the washing steps to be performed at 150 to 200°C in order to provide good extraction of the salt (column 5, lines 6 to 16). At lines 16 to 18 it is stated that - "The washing and filtration can be conducted batchwise or continuously". That is the first and last mention of the possibility of continuous operation, the two examples being confined to batch operations.

5. *Problem and its solution*

5.1 The problem with which the application in suit is concerned is the attainment of a process for the removal to a satisfactory degree of occluded salt in the final stage of PPS production which saves both time and energy, these being advantages which are attainable over the methods described in detail in document (1). Although the data given in Table I of the application in suit are nothing like as detailed as might be desired, in that there is no direct comparison of those known methods with the claimed continuous process, the washing

in both cases being continued for the **same period of time**, so that a difference in salt removal could be observed, or both carried out to the **same level of salt removal**, so that it could be seen that the same effect is achieved in a shorter time interval, nevertheless the existence of some improvement is sufficiently plausible to be accepted, in the present absence of any evidence to the contrary.

5.2 Continuous runs 2 and 3 in Table I of the application in suit show that in 1.5 minutes at 185°C the ash content was reduced to 0.94 and 0.92%. Batch run 6 shows that at 182°C, in a time said to be 5 to 10 minutes, the ash level was reduced to 0.80 to 0.83%. It is to be observed that all three variables, i.e. time, temperature, and degree of salt removal, are different, although the temperatures and degrees of salt removal are only different to a small extent. The application in suit states at page 5, line 34 to page 6, line 3 that the salt extraction according to the continuous process was considerably faster, and thus more efficient, than a batch process conducted at a comparable temperature.

5.3 In the Board's view, the above-referred results barely suffice to support that conclusion. Against the paucity of demonstrated results, the Board finds it inherently credible that when it comes to salt removal by leaching with hot water, the use of a higher temperature, which is economically attainable through continuous washing involving heat recuperation, is likely to result in a faster process, coupled with a saving in energy consumption. The Board is therefore satisfied that the process disclosed in accordance with the alleged invention affords a credible solution to the above defined problem.

6. *Inventive step*

6.1 The issue of existence of any inventive step turns on whether a skilled person, having as his starting point the disclosure of document (1), and confronted with the problem of saving both time and energy in the final salt removal stage of the PPS production process, would have seen in its isolated reference, without any further detail, to a continuous process at column 5, line 17 any real inducement to adopt the claimed continuous process. In the view of the Board, the mere reference to that possibility in document (1), taken in the context of that document where only batch processes are described in detail, and also taken in the context of an industry in which, on the available evidence, batch processes were invariably used, seems to the Board to advance the teaching of a continuous process in document (1) very little further than the known desirability of using continuous washing processes, which is ever-present in the mind of skilled workers.

6.2 In contrast to the view of the decision under appeal, at this stage of proceedings, when such evidence as there is remains uncontested, the Board is prepared to attach decisive significance to the evidence of Mr Scoggins (see paragraph III above) which is to the following effect:

- (a) Since the start of PPS production in 1972, continuous processes for the removal of salt have been in contemplation, but not adopted.
- (b) Batch processes lend themselves to efficient stirring of the PPS-water slurry, and afford the opportunity for extended contact time between water and PPS, both tending to assist in the removal of occluded salt.

(c) It was feared that the above mentioned advantages could be lost if a continuous process were to be adopted, and there was a genuine fear that, due to the fine particulate nature of the polymer, there would be serious problems resulting from pipe blockages.

6.3 Taking into account the general desirability of replacing batch processes with continuous ones in the interest of economy, the Board considers that the fact that ten years passed between the start of production of PPS in 1972, to the priority date of the present application in 1982, suggests that it was not obvious to go over to the claimed continuous process. If the "prejudice" referred to by Mr Scoggins had been more imaginary than real, the Board would have expected such continuous process to have been adopted sooner. In the light of the information at present available to it, consisting of a limited amount of prior art and the unchallenged statement of Mr Scoggins, the Board is prepared to accept for the present at face value his statement that there was a significant prejudice against the use of such continuous process, due to fears of pipe blockage, which later proved to have been unjustified. Accordingly the Board is satisfied that the process as now defined by Claim 1 was not obvious.

7. *Conclusion*

The subject-matter of Claim 1 of the patent in issue thus involves an inventive step as required by Article 56 EPC, and is therefore patentable. The same applies to the dependent Claims 2 to 7 which relate to modifications of the process falling wholly within the scope of Claim 1.

A patent cannot yet be granted, however, because the description still requires adaptation to the claims allowed. This would include conversion of weights and measures into the metric system. For that purpose the case has to be remitted to the Examining Division (Article 111 EPC).

Order

For these reasons, it is decided that:

1. The decision under appeal is set aside.
2. The case is remitted to the Examining Division with the order that a patent be granted on the basis of Claims 1 to 5, referred to in paragraph II above, subject to appropriate adaptation of the description.

The Registrar:



E. Gorgmaier

The Chairman:



F. Antony