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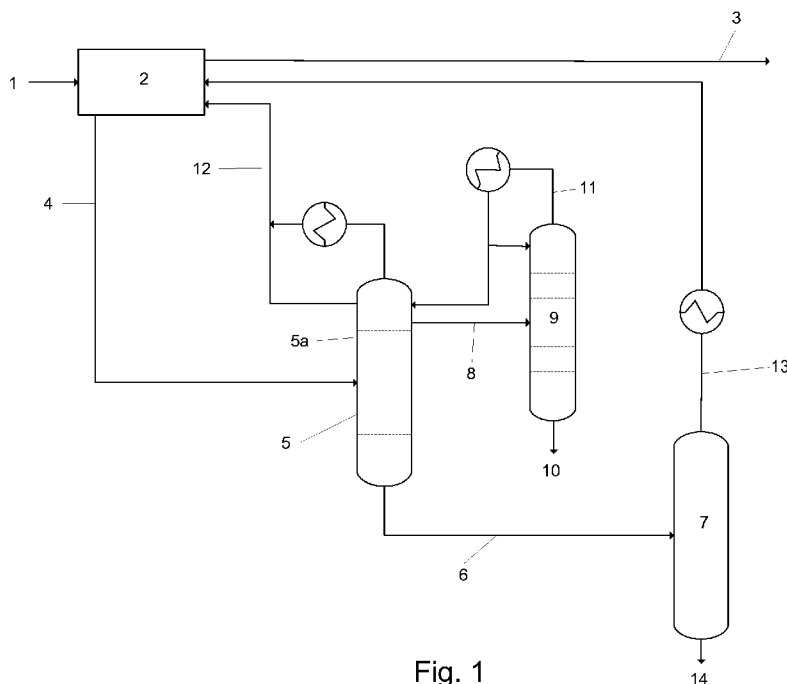
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[Continued on next page]

(54) Title: PROCESS FOR SEPARATING PHENOLS AND BTX AROMATICS FROM GAS CONDENSATE



(57) Abstract: A process and plant for separating phenols from gas condensate of the fixed-bed gasification of coal by extraction with an organic solvent, wherein the solvent is circulated and wherein the phenols are separated from the loaded solvent by distillation, and wherein in an additional distillation step aromatic hydrocarbons, such as benzene, toluene and xylenes (BTX aromatics) are separated from the solvent.

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SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, **Published:**
GW, KM, ML, MR, NE, SN, TD, TG).

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10 **Process for Separating Phenols and BTX Aromatics from Gas Condensate**

Field of the Invention

15 The invention relates to a process for separating phenols and BTX aromatics (benzene, toluene, xylenes) from the gas condensate of the fixed-bed gasification of coal by extraction with an organic solvent, comprising the following steps:

20 a) supplying a gas condensate stream loaded with phenols and BTX aromatics from the gas condensate of the fixed-bed gasification of coal to an extraction zone,

25 b) extraction of the gas condensate stream loaded with phenols and BTX aromatics in the extraction zone with an organic solvent, wherein a gas condensate stream depleted of phenols and BTX aromatics is obtained as raffinate and the solvent loaded with phenols and BTX aromatics is obtained as extract,

30 c) supplying the extract to a first distillation and distillative separation into a first bottom product containing phenols and the solvent and into a first overhead product containing the BTX aromatics and the solvent, wherein the first overhead product is recirculated to the extraction zone,

 d) supplying the first bottom product containing phenols and the solvent to a second distillation and distillative separation into a second bottom product

containing phenols and a second overhead product containing the solvent, wherein the second overhead product is recirculated to the extraction zone.

The invention likewise relates to a plant for operating the process.

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Prior art

In the fixed-bed pressure gasification of carbonaceous fuels, such as coke or coal, an aqueous so-called gas condensate among other things is obtained as by-product. A frequently used fixed-bed pressure gasification process is the so-called
10 Sasol-Lurgi dry bottom gasification process (FBDB process) which is described in the textbook GASIFICATION, 2nd Edition, Christopher Higman, Maarten van der Burgt, Gulf Professional Publishing. Fig. 5.3 on page 97 reveals how the gas condensate (gas liquor) is obtained by condensing out of the raw gas.

15 Before this gas condensate can be transferred to a biological clarification plant, some ingredients must be removed therefrom, in particular tars, oils, phenols and gases, such as ammonia, hydrogen sulfide and carbon dioxide. The basic sequence of these cleaning steps is described in Ullmann's Encyclopedia of Industrial Chemistry, 6th edition, vol. 15, page 437. Known treatment processes
20 for the gas condensate in this connection are the Phenosolvan process and the CLL process.

The separation of the phenols from the gas condensate frequently is effected by extraction with an organic solvent, as it is described in Ullmann's Encyclopedia of
25 Industrial Chemistry, 6th edition, vol. 15, on page 433, Fig. 71. The extraction device frequently is designed according to the so-called mixer-settler principle. This is a continuous process, in which the loaded extracting agent constantly is circulated via a distillation plant in which the phenols are separated from the loaded extracting agent by distillation. A frequently used solvent or extracting
30 agent is diisopropyl ether (DIPE). In this distillation, multistage in many cases, a phase rich in phenols is obtained as bottom product and discharged from the process or supplied to the further treatment for obtaining the phenols, while the

solvent largely liberated from phenols is obtained as overhead product and again supplied to the extraction process as extracting agent.

5 Beside the phenols, benzene, toluene and xylenes (BTX aromatics) from the gas condensate also are absorbed by the extracting agent during the extraction. In the existing process it is disadvantageous that these substances cannot be separated by the existing distillation treatment and thus are enriched in the extracting agent. The extraction properties of the solvent used are negatively influenced thereby and the effectiveness of the extraction process thus is deteriorated. When a
10 sufficient effectiveness of the process no longer is given, the solvent used as extracting agent must be discarded and replaced.

15 Therefore, it is the object of the invention to provide a process for separating phenols from gas condensate, which avoids the disadvantages described above.

Description of the Invention

The object is solved by a process according to the features of claim 1 and by a plant with the features of claim 7.

Process according to the invention:

20 A process for separating phenols and BTX aromatics (benzene, toluene, xylenes) from the gas condensate of the fixed-bed gasification of coal by extraction with an organic solvent, comprising the following steps:

25 a) supplying a gas condensate stream 1 loaded with phenols and BTX aromatics to an extraction zone 2,

30 b) extraction of the gas condensate stream loaded with phenols and BTX aromatics in the extraction zone 2 with an organic solvent, wherein a gas condensate stream 3 depleted of phenol and BTX aromatics is obtained as raffinate and the solvent 4 loaded with phenols and BTX aromatics is obtained as extract,

c) supplying the extract 4 to a first distillation 5 and distillative separation into a first bottom product 6 containing phenols and the solvent and into a first overhead

product 12 containing the BTX aromatics and the solvent, wherein the first overhead product is recirculated to the extraction zone,

d) supplying the first bottom product 6 containing phenols and the solvent to a second distillation 7 and distillative separation into a second bottom product 14

5 containing phenols and a second overhead product 13 containing the solvent, wherein the second overhead product is recirculated to the extraction zone 2, characterized in that from the first distillation a partial stream 8 is discharged, which contains BTX aromatics and the solvent, wherein the partial stream is supplied to a third distillation 9 and by distillation is separated into a third bottom
10 product 10 containing BTX aromatics and a third overhead product 11 containing the solvent, wherein the third bottom product 10 containing BTX aromatics is discharged and the third overhead product 11 containing the solvent is recirculated to the extraction zone 2.

15 Plant according to the invention:

A plant for separating phenols and BTX aromatics (benzene, toluene, xylenes) from the gas condensate 1 of the fixed-bed gasification of coal, comprising

- an extraction zone 2 for obtaining a gas condensate stream 3 depleted of phenols and BTX aromatics as raffinate and a solvent stream 4 loaded with
20 phenols and BTX aromatics as extract,
- a first distillation column 5 for separating the extract into a first bottom product 6 containing phenols and the solvent and into a first overhead product 12 containing BTX aromatics and the solvent,
- a conduit for recirculating the first overhead product to the extraction zone,
- 25 - a second distillation column 7 for separating the first bottom product into a second bottom product 14 containing phenols and a second overhead product 13 containing the solvent,
- a conduit for recirculating the second overhead product to the extraction zone,
- a conduit 8 for discharging a partial stream containing BTX aromatics and the
30 solvent from the first distillation column,

- a third distillation column 9 for separating the partial stream into a third bottom product 10 containing BTX aromatics and a third overhead product 11 containing the solvent,
- a conduit for recirculating the third overhead product 11 to the extraction zone 2.

5

It is a particular advantage of the invention that merely a partial stream is withdrawn from the first distillation for removing BTX aromatics, which is particularly favorable for the economy of the process. Furthermore, for restoring the extraction properties of the solvent used it mostly is not necessary to completely remove the BTX aromatics from the same. In this way, the sizes of the apparatuses used can be kept small and the energy consumption can be minimized when carrying out the process according to the invention.

10

Preferred Aspects of the Invention

15 A preferred aspect of the invention consists in that the partial stream 8 is discharged from the enriching section 5a of the first distillation and the third overhead product 11 is recirculated into the enriching section of the first distillation and via the same is recirculated to the extraction zone 2. In this way, the transport of the partial stream between the distillation columns can be effected by

20 a suitable adjustment of a pressure difference between the columns and by gravitation, so that additional conveying means, such as pumps, can be omitted. Furthermore, the heat content of the partial stream 8 is optimally utilized, so that the required reboiler performance for the third distillation 9 can be reduced.

20

25 A further preferred aspect of the invention consists in that diisopropyl ether (DIPE) is used as organic solvent. This solvent has long since proven successful for this extraction process and is available at low cost. In this connection it is particularly advantageous when the concentration of the BTX aromatics in the solvent recirculated to the extraction zone 2 is below 50 wt-%, preferably below 20 wt-%.

30 At these concentrations, the required extraction properties of the solvent are restored sufficiently, but at the same time the required separation effort for the distillative separation of the BTX aromatics from the solvent is kept small.

30

A further preferred aspect of the invention consists in that the extraction is carried out by means of a mixer-settler system. The same likewise have long since proven successful for this process. They can be produced at low cost and are easy to maintain.

5

A further preferred aspect of the invention consists in that the third distillation is designed as distillation column and the partial stream is charged approximately in the middle of this column, so that the enriching section and the stripping section each have the same number of trays. For the present separation task these process components and this procedure have long since proven quite successful.

10

A preferred aspect of the plant according to the invention consists in that it comprises a conduit for recirculating the third overhead product to the enriching section of the first distillation column, wherein the conduit for discharging the partial stream containing BTX aromatics and the solvent also is mounted on the enriching section of the first distillation column.

15

Exemplary embodiment

Further features, advantages and possible applications of the invention can also be taken from the following description of an exemplary embodiment and the drawing. All features described and/or illustrated form the subject-matter of the invention per se or in any combination, independent of their inclusion in the claims or their back-reference.

20

With reference to the drawing, Fig. 1, the process according to the invention will now be explained in detail. In the drawings:

25

Fig. 1 shows a schematic flow diagram of the process according to the invention.

30

In the process flow diagram schematically shown in Fig. 1, the gas condensate 1 liberated from tar and oil is introduced into the extraction zone 2. The gas

condensate largely liberated from phenols and BTX aromatics leaves the extraction plant as raffinate stream 3 and is supplied to the further treatment not shown in Fig. 1.

5 The organic extracting agent 4 loaded with phenols and BTX aromatics, here diisopropyl ether (DIPE), is charged to the first distillation column 5. As bottom product 6 a phase rich in phenols is obtained, which is still loaded with residual amounts of DIPE and is charged to a second distillation column 7. From the enriching section 5a of the first distillation column 5 a stream 8 is withdrawn from
10 the third theoretical tray (counting the column trays from top to bottom) and charged to a third distillation column 9, which is designed as column with 30 theoretical trays. The stream 8 is charged onto the tray 17 of the column 9.

In the third distillation column 9 a phase rich in BTX aromatics is separated from
15 the stream 8 as bottom product 10. The overhead product 11 of column 9, the extracting agent DIPE largely liberated from BTX aromatics, is condensed by a water cooler and as return flow partly recirculated to the first tray of the column 9. Another part of the overhead product 11 is guided back into the enriching section 5a of the first column 5. The overhead product 12 of the column 5, the extracting
20 agent DIPE largely liberated from phenols and BTX, finally is returned into the extraction zone 2 and again used there for the extraction.

In the second distillation column 7, residual amounts of DIPE are separated from the phase 6 rich in phenols as overhead product 13 and likewise recirculated into
25 the extraction plant for reuse as extracting agent. As bottom product 14 a phase rich in phenols is obtained, which largely is liberated from extracting agent and also is referred to as Crude Tar Acid (CTA). This phase rich in phenols possibly can be supplied to the further treatment for obtaining the phenols as valuable product. Furthermore, the CTA also can be used as washing agent, for example in
30 an acid gas absorber (not shown in Fig. 1).

Industrial Applicability

The invention effects a longer usability of the extracting agent with constant extraction efficiency and hence improves the economy of the extraction process.

5

List of Reference Numerals

- | | | |
|----|----|---|
| 10 | 1 | gas condensate |
| | 2 | extraction zone |
| | 3 | gas condensate, depleted of phenols and BTX aromatics |
| | 4 | extracting agent, loaded with phenols and BTX aromatics |
| | 5 | first distillation |
| 15 | 6 | first bottom product, phase rich in phenols with residues of extracting agent |
| | 7 | second distillation |
| | 8 | partial stream, loaded with BTX aromatics |
| | 9 | third distillation |
| | 10 | third bottom product, phase rich in BTX aromatics |
| 20 | 11 | third overhead product, depleted of BTX aromatics |
| | 12 | first overhead product, extracting agent, largely liberated from phenols and BTX aromatics |
| | 13 | second overhead product, extracting agent, largely liberated from phenols and BTX aromatics |
| 25 | 14 | second bottom product, phase rich in phenols |

Claims:

1. A process for separating phenols and BTX aromatics (benzene, toluene,
xylenes) from the gas condensate of the fixed-bed gasification of coal by
5 extraction with an organic solvent, comprising the following steps:
a) supplying a gas condensate stream 1 loaded with phenols and BTX
aromatics to an extraction zone 2,
b) extraction of the gas condensate stream loaded with phenols and BTX
aromatics in the extraction zone 2 with an organic solvent, wherein a gas
10 condensate stream 3 depleted of phenol and BTX aromatics is obtained as
raffinate and the solvent 4 loaded with phenols and BTX aromatics is
obtained as extract,
c) supplying the extract 4 to a first distillation 5 and distillative separation
into a first bottom product 6 containing phenols and the solvent and into a
15 first overhead product 12 containing the BTX aromatics and the solvent,
wherein the first overhead product is recirculated to the extraction zone,
d) supplying the first bottom product 6 containing phenols and the solvent
to a second distillation 7 and distillative separation into a second bottom
product 14 containing phenols and a second overhead product 13
20 containing the solvent, wherein the second overhead product is recirculated
to the extraction zone 2,
characterized in that from the first distillation a partial stream 8 is
discharged, which contains BTX aromatics and the solvent, wherein the
partial stream is supplied to a third distillation 9 and by distillation separated
25 into a third bottom product 10 containing BTX aromatics and a third
overhead product 11 containing the solvent, wherein the third bottom
product 10 containing BTX aromatics is discharged and the third overhead
product 11 containing the solvent is recirculated to the extraction zone 2.
2. The process according to claim 1, characterized in that the partial stream 8
30 is discharged from the enriching section 5a of the first distillation and the
third overhead product 11 is recirculated into the enriching section 5a of the
first distillation and via the same is recirculated to the extraction zone 2.

3. The process according to claim 1 or 2, characterized in that diisopropyl ether is used as organic solvent.
4. The process according to claim 3, characterized in that the concentration of the BTX aromatics in the solvent recirculated to the extraction zone 2 is
5 below 50 wt-%, preferably below 20 wt-%.
5. The process according to any of the preceding claims, characterized in that the extraction is carried out by means of a mixer-settler system.
6. The process according to any of the preceding claims, characterized in that
10 the third distillation is designed as distillation column and the partial stream is charged approximately in the middle of this column.
7. A plant for separating phenols and BTX aromatics (benzene, toluene, xylenes) from the gas condensate 1 of the fixed-bed gasification of coal, comprising
 - an extraction zone 2 for obtaining a gas condensate stream 3 depleted of
15 phenols and BTX aromatics as raffinate and a solvent stream 4 loaded with phenols and BTX aromatics as extract,
 - a first distillation column 5 for separating the extract into a first bottom product 6 containing phenols and the solvent and into a first overhead product 12 containing BTX aromatics and the solvent,
 - 20 - a conduit for recirculating the first overhead product to the extraction zone,
 - a second distillation column 7 for separating the first bottom product into a second bottom product 14 containing phenols and a second overhead product 13 containing the solvent,
 - 25 - a conduit for recirculating the second overhead product to the extraction zone,
 - a conduit 8 for discharging a partial stream containing BTX aromatics and the solvent from the first distillation column,
 - 30 - a third distillation column 9 for separating the partial stream into a third bottom product 10 containing BTX aromatics and a third overhead product 11 containing the solvent,

- a conduit for recirculating the third overhead product 11 to the extraction zone 2.

- 5 8. The plant according to claim 7, furthermore comprising a conduit for recirculating the third overhead product to the enriching section of the first distillation column, wherein the conduit for discharging the partial stream containing BTX aromatics and the solvent also is mounted on the enriching section of the first distillation column.

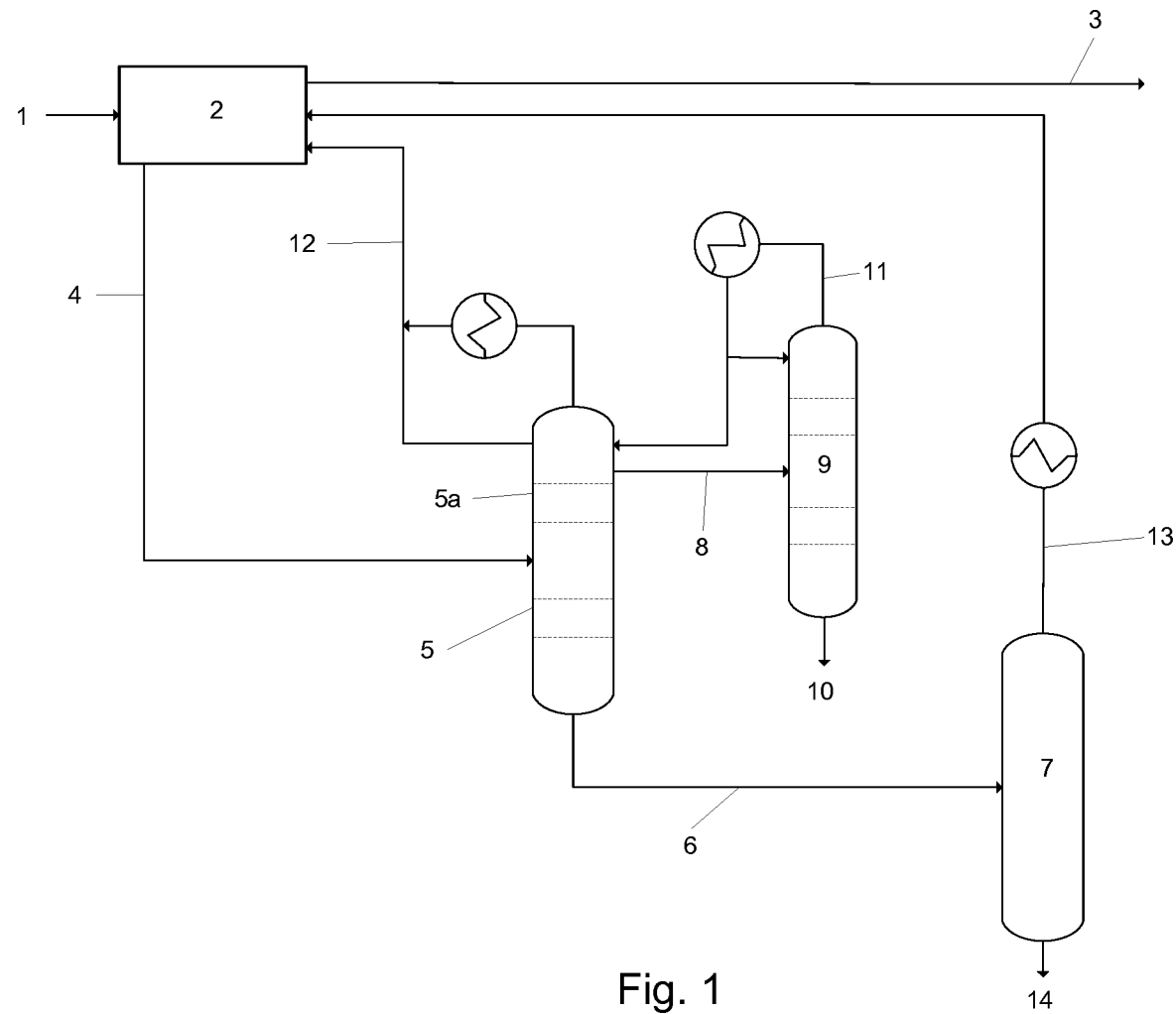


Fig. 1

INTERNATIONAL SEARCH REPORT

International application No
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A. CLASSIFICATION OF SUBJECT MATTER
INV. B01D11/04 C07C7/04 C10G53/04 C10K1/00 C10G21/00
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
B01D C07C C10G C10K

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 5 716 524 A (STOENNER HANS-MARTIN [DE] ET AL) 10 February 1998 (1998-02-10) column 2, line 17 - line 20; figures 1-2 -----	1-8
A	US 2013/225838 A1 (LEE FU-MING [US] ET AL) 29 August 2013 (2013-08-29) figures 1-4 -----	1-8
A	US 2 794 839 A (BROUGHTON DONALD B) 4 June 1957 (1957-06-04) figure 1 -----	1-8



Further documents are listed in the continuation of Box C.



See patent family annex.

* Special categories of cited documents :

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2015/025046

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