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(54) Title: REMOVAL OF HYDROGEN IN A SIDE STREAM

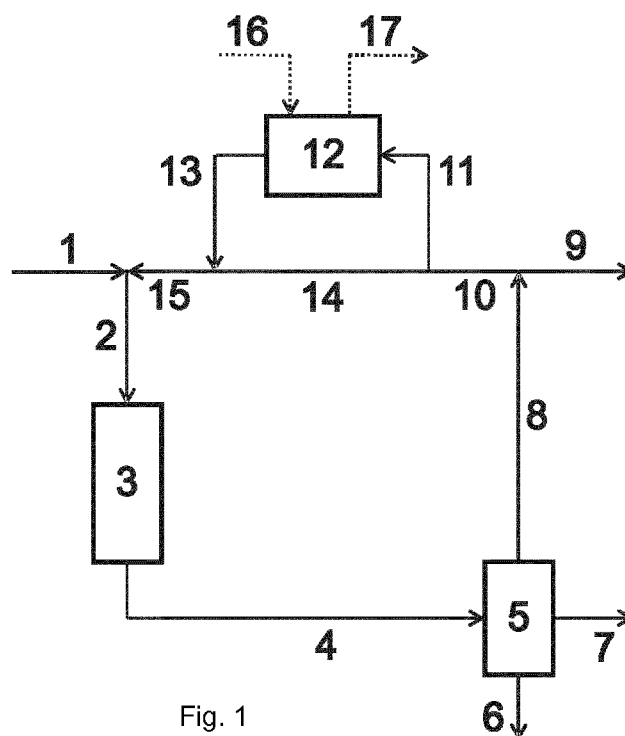


Fig. 1

(57) Abstract: The present application relates to a process for conversion of methanol to hydrocarbons comprising aromatics (MTA), said process comprising the steps of - reacting a reactor feed comprising methanol over a bifunctional catalyst thereby obtaining a reaction effluent, - separating the reaction effluent into at least an aqueous condensate stream, a liquid hydrocarbon stream and a gas stream, - obtaining a first recycle stream from the gas stream - obtaining a first side stream from the gas stream, - passing the first side stream through at least one hydrogen (H₂) removal step thereby obtaining a hydrogen depleted stream, and - mixing the first recycle stream and the hydrogen depleted stream with a first feed stream comprising methanol to obtain the reactor feed.



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MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM,
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Declarations under Rule 4.17:

- *as to the identity of the inventor (Rule 4.17(i))*
- *as to applicant's entitlement to apply for and be granted a patent (Rule 4.17(ii))*
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Title: Removal of hydrogen in a side stream

Since its discovery in the 1970's, zeolite catalyzed conversion of methanol to hydrocarbons has become increasingly important in the chemical industry and several variations of the process have been commercialized including MTO (methanol-to-olefins), MTP (methanol-to-propylene), and MTG (methanol-to-gasoline). Herein, the focus is on the MTA (methanol-to-aromatics) process, in which methanol is converted over a metal/oxide containing zeolite catalyst to a mixture of hydrocarbons preferably with a high content of aromatic compounds. In methanol to aromatics (MTA) processes the yield and product composition may be controlled by choice of e.g. catalyst and process conditions as well as the composition of the feed stream and recycles.

In the MTA process accumulation of hydrogen in the recycle may inhibit dehydrogenation reactions taking place over the MTA catalyst resulting in lower yield of aromatics. Therefore, it may be beneficial to remove hydrogen from the recycle stream. Several methods can be used for removal of hydrogen, such as including a perm-selective membrane and/or by selective oxidation in the recycle stream.

The present invention address some of the challenges in known setups for removal of hydrogen in recycles. According to the present process is provided a process which reduces the energy consumption and/or hardware requirements related to the control of the composition of one or more recycled streams.

These and other advantages are provided by a process for conversion of methanol to hydrocarbons comprising aromatics (MTA), said process comprising the steps of

- reacting a reactor feed stream comprising methanol and/or dimethylether (DME) over a bifunctional catalyst thereby obtaining a reactor effluent,
- separating the reactor effluent into at least an aqueous condensate stream, a liquid hydrocarbon stream and a gas stream,
- obtaining a first recycle stream from the gas stream
- obtaining a at least a first side stream from the gas stream,
- passing the first side stream through at least one hydrogen (H₂) removal step thereby obtaining a hydrogen depleted stream, and

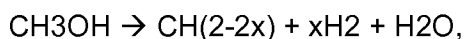
- mixing the first recycle stream and the hydrogen depleted side stream with a first feed stream comprising methanol to obtain the reactor feed.

5 In the MTA conversion process methanol is converted into aromatics while dehydrogenation of hydrocarbons including one or more types of hydrocarbons, comprising naphthenes, paraffins and isoparaffins, into olefins and/or aromatics is taking place.

10 The MTA conversion process may preferably be carried out at a pressure 5-60 bar, preferably 10-40 bar, temperature 300-500°C, preferably 330-480°C and/or weight hourly space velocities (kg alcohol and/or ether feed per kg of catalyst per hour) between 0.1 and 10 preferably 0.3-3.

15 The MTA process may provide a product particularly suited as feedstock for downstream aromatics processing, e.g. for making para-xylene. The product may also provide useful as a high-octane blendstock for gasoline manufacture in refineries.

20 As opposed to traditional methanol-to-hydrocarbons reactions (MTG and MTO) this invention provides a hydrocarbon product which has an overall H/C ratio of less than two, according to



25 thereby forming more aromatics on the expense of n- and i-paraffins, in particular, light n- and i-paraffins, such as ethane, propane and butanes, and providing a highly aromatic hydrocarbon product.

30 The obtained hydrocarbon product of the present invention may be rich in aromatics and provide useful as feed or aromatics processing, as blendstock, e.g. similar to reformate, in refineries, or the product or part of the product may be upgraded by conventional means to provide a finished gasoline product.

The separation step, where the effluent from the reaction step is separated into a gas phase and aqueous and hydrocarbon liquid phases, is typically conducted by cooling the conversion effluent essentially at the synthesis pressure, at 10-50 bar, typically by cooling to a temperature between 25 and 60°C.

5

The liquid hydrocarbon phase may comprise various aromatics such as benzene, toluene, xylenes, ethylbenzene, and heavier aromatic compounds with 9 or more carbon atoms, as well as paraffins, isoparaffins, olefins, and naphthenes. Most of the hydrocarbons present in the liquid hydrocarbon stream may be components with 4 or more carbon atoms, but this stream may also comprise lighter hydrocarbons in low concentrations. The liquid hydrocarbon stream may also comprise small amounts of dissolved gases such as CO₂, CO, C1-C4 hydrocarbons, and H₂.

10

The gas stream comprises mainly light hydrocarbons such as methane, ethane, propane, butane, ethylene, and/or propylene, as well as carbon oxides, carbon dioxide, and/or hydrogen.

15

The aqueous condensate comprises mainly water, but may also comprise small amounts of various oxygenates including methanol, other alcohols, aldehydes, and ketones, hydrocarbons as well as dissolved gases.

20

Methanol-to-hydrocarbons processes are generally very exothermic, and therefore means need to be taken to control the temperature of the reactor. One method used in the industry is to include a recycle over the reactor which acts as a heat sink, allowing for control of the exit temperature of the reactor by adjusting the recycle/feed ratio. In the MTA process, H₂ will accumulate in the recycle, inhibiting the dehydrogenation reactions over the MTA catalyst resulting in low yields of aromatics. Therefore, it may be beneficial to remove H₂ from the recycle stream.

25

In this invention H₂ is removed from at least one side stream rather than the full recycle stream. In the MTA process, the recycle stream is typically very large and it generally requires large equipment and significant amounts of energy to remove hydrogen from the full recycle stream. By using the process described here, a compromise between the size of the side stream and the degree of hydrogen removal in the side stream may result in smaller equipment size (and capital costs), as well as lower energy consumption.

In the present process it is also possible to have more than one side stream for example to reduce the size of each side stream and/or in order to remove H₂ by different means thereby controlling the composition of the sum of the H₂ depleted streams even further.

The recycle and hydrogen depleted side stream(s) may be mixed together before being added to the first feed stream or may be added individually to the first feed stream and/or the reactor to form the reactor feed. They may also be added to the reactor in the form of a quench stream.

Several methods can be used for removal of H₂ including a selective membrane and/or by selective oxidation (PrOx) of H₂. However, this invention opens the possibility to use less complex methods, such as full oxidation of the side stream. In this case, the degree of hydrogen removal from the reactor loop is controlled by adjusting the size of the side stream and/or the degree of oxidation of the side stream.

The degree of H₂ removal affects the products from the reaction. If no hydrogen is removed from the system, hydrogen will accumulate in the loop and, eventually, the dehydrogenation reaction will cease. Therefore, continuous removal of hydrogen from the loop may be required in order to sustain the dehydrogenation reaction at a sufficient level.

In case the H₂ depletion generates heat (e.g. as in combustion reactions), the heat released by the combustion of the side stream may be used internally in the plant or exported (e.g. steam production).

Preferably the first feed stream comprises 50 – 100 mol% methanol and/or DME, such as 90 – 100 mol% methanol and/or DME. The feed stream may contain various amounts of water.

- 5 Preferably the reactor feed comprises 0.5 – 50 mol% methanol and/or DME, such as 2 – 25 mol% methanol and/or DME, or more preferably 5 – 20 mol% methanol and/or DME.

- 10 Preferably the reactor feed stream comprises 0 – 20 mol% H₂, such as 0 – 10 mol% H₂, or more preferably 0 – 5 mol% H₂, or more preferably 0 – 2 mol% H₂.

The reactor feed stream may further comprise CO, CO₂, methane, ethane, propane, ethylene, propylene, and/or higher hydrocarbons.

- 15 In advantageous embodiments the ratio of the first recycle stream/side streams is 0.2 – 50, such as 1 – 10 in order to obtain a sufficient overall removal of H₂ removal while still keeping the size of the equipment as well as the operating costs related to removal of H₂ in the side stream down.

- 20 The size of the recycle stream (sum of first recycle and effluent from H₂ removal unit(s)) may need to be sufficiently high in order to be able to control the temperature in the MTA reactor. For example the ratio first feed stream/recycle is 0.01 – 2, such as 0.02 – 1 or more preferable 0.05 – 0.5.

- 25 In order to achieve a desired selectivity to aromatics, a MTA catalyst preferably comprises a zeolite or zeotype as well as a metal/oxide function. The zeolite/zeotype is responsible for conversion of oxygenates to hydrocarbons, while the metal/oxide function is responsible for dehydrogenation of intermediate hydrocarbons, e.g. dehydrogenation of naphthenes to aromatics and/or dehydrogenation of paraffins to olefins. The combination of a zeolite function and a dehydrogenation function is essential for achieving a
30 high yield of aromatics in the MTA process.

Different zeolite/zeotypes may be employed, including ZSM-5, ZSM-11, ZSM-23, ZSM-48, SAPO-34, however ZSM-5 may be preferred, since it has a suitable size selectivity

to the desired methylated monocyclic aromatic species as well as a relatively low coking rate. The metal component of the MTA catalyst may in advantageous embodiments be chosen from Zn, Ga, In, Ge, Ni, Mo, P, Ag, Sn, Pd and Pt or combinations thereof. Zn may be preferred over the other metals. Thus, a catalyst comprising Zn/ZSM-5 may be a particularly preferred catalyst system for the MTA process. Furthermore, the MTA catalyst may comprise phosphorus, which leads to better hydrothermal stability of the catalyst and thus longer ultimate catalyst lifetime.

It may be preferred to use a binder material in order to shape the catalyst. This binder material may be a normally employed binder material such as Al₂O₃, MgAl₂O₄, SiO₂, ZrO₂, TiO₂, MgO or mixtures thereof. Al₂O₃ may be preferred. If Al₂O₃ is used as binder, Zn may be present in the catalyst as ZnAl₂O₄. Similarly, if Al₂O₃ is used as binder P may be present in the catalyst as AlPO₄.

An MTA catalyst may comprise 0.2 – 15 wt% Zn, or more preferably 3 – 15 wt% Zn or even more preferably 5 – 15 wt% Zn. Furthermore, an MTA catalyst may comprise 0 – 10 wt% P, or more preferably 0.1 – 8 wt% P or even more preferably 0.5 – 5 wt% P.

The MTA process may be carried out in one or more fixed bed and/or fluid bed reactors.

The process may be carried out in a plant comprising a first feed stream, a reactor, a separator for separating a reaction effluent into at least an aqueous condensate stream, a liquid hydrocarbon stream and a gas stream,

- means for recycling a first recycle stream from the gas stream to the reactor
- means for obtaining a first side stream from the gas stream,
- means for removing H₂ from the first side stream to obtaining a hydrogen depleted stream, and
- means for adding the hydrogen depleted stream to the reactor,
- optionally means for purging at least part of the first recycle stream,

The recycle and hydrogen depleted side stream(s) may be mixed together before being added to the first feed stream or may be added individually to the first feed stream and/or the reactor.

The invention is illustrated with reference to the accompanying drawings 1 – 3. The drawings are not to be construed as limiting to the invention.

5 Fig. 1 shows a layout with a feed stream comprising methanol (1) which is mixed with a recycle stream (15) to obtain the reactor feed stream (2) entering the MTA reactor (3). The reactor effluent (4) enters a separator (5) which separates the reactor effluent into an aqueous condensate (6), a liquid hydrocarbon (product) stream (7), and a gas stream (8). Part of said gas stream (8) is purged (9) and the remaining stream (10), is
10 split such that part of it, called the first side stream (11), is sent to the hydrogen removal unit (12), while the remainder, called the first recycle stream (14), is allowed to bypass the hydrogen removal unit. The hydrogen depleted stream (13) leaving the hydrogen removal unit is mixed with the first recycle stream 14 to form the recycle stream (15). Depending on the nature of the hydrogen removal unit, a stream entering the hydrogen removal unit (16) and/or a stream leaving the hydrogen removal unit (17) may
15 be required.

The hydrogen depleted stream (13) may be mixed with the First recycle stream (14) and/or Feed stream (1) in various point upstream the reactor (3) and/or in the reactor.

20

The hydrogen removal unit may be any piece of equipment/reactor/process which results in a decrease in the concentration of hydrogen, i.e. stream 13 has a lower hydrogen concentration than stream 11. The hydrogen removal unit may for instance be a membrane, a PrOx reactor, a methanation reactor, a methanol synthesis reactor or a
25 (catalytic) oxidation reactor.

If a hydrogen selective membrane is used, the size of the membrane is to a large extent determined by the size of stream 11. By having a relatively small first side stream 11 (low 11/14 ratio), a relatively small membrane may be used resulting in significantly
30 lower capital expenses. Furthermore, a smaller compressor for re-pressurizing the retentate (stream 13) to loop pressure is required, resulting in lower capital expenses as well as lower operating costs. When a selective membrane is used, stream 17 represent the H₂ rich permeate, which is removed from the system.

If a PrOx reactor, methanation reactor or methanol synthesis reactor is used, the size and process conditions of the reactor can be optimized by adjusting the size of the side stream (11/14 ratio). Thus a more efficient overall process can be obtained.

5 Hydrogen removal in the side stream can also be done by simple oxidation, meaning that an amount of O₂ (or air or partially oxygen depleted air) is added leading to conversion of H₂ as well as CO and hydrocarbons to H₂O and CO₂. The degree of hydrogen removal from the loop is largely determined by the size of the side stream (11/14 ratio), but the amount of oxygen added can also be adjusted to control the amount of
10 H₂ removed from the stream. The size of stream 16 (16/11 ratio) as well as the oxygen concentration in stream 16 determines the amount of H₂ (and CO) removed from stream 11. Preferably, the H₂ concentration in stream 13 is low (ultimately below 0.1 mol%) The oxidation reactor may be a catalytic oxidation reactor. Other oxidants than O₂ may be applied, for instance H₂O₂.

15

Fig. 2 shows another embodiment, where stream 11 is a side stream taken from stream 9:

Fig. 3 shows yet another embodiment, wherein stream 11 is a side stream taken from stream 8.

20

Claims

1. A process for conversion of methanol to hydrocarbons comprising aromatics (MTA), said process comprising the steps of
- 5 - reacting a reactor feed comprising methanol and/or DME over a bifunctional catalyst thereby obtaining a reaction effluent,
- separating the reaction effluent into at least an aqueous condensate stream, a liquid hydrocarbon stream and a gas stream,
- obtaining a first recycle stream from the gas stream
- 10 - obtaining a first side stream from the gas stream,
- passing the first side stream through at least one hydrogen (H₂) removal step thereby obtaining a hydrogen depleted stream, and
- mixing the first recycle stream and the hydrogen depleted stream with a first feed stream comprising methanol and/or DME to obtain the reactor feed.
- 15
2. Process according to claim 1, wherein the hydrogen removal step comprises a membrane, a PrOx reactor, a methanation reactor, a methanol synthesis reactor, oxidation reactor or a catalytic oxidation reactor or combinations thereof.
- 20
3. Process according to any of the preceding claims wherein, the H₂ content in the hydrogen depleted steam is adjusted by controlling the amount of oxidant added to the oxidation step.
- 25
4. Process according to any of the preceding claims wherein heat generated by H₂ removal is used in the up or downstream the MTA process and or is exported e.g. for steam production.
5. Process according to any of the preceding claims, wherein the ratio of the First recycle stream/Side stream is 0.2 – 50, such as 1 – 10.
- 30
6. Process according to any of the preceding claims, wherein the ratio of the First feed stream/First recycle+Side stream is 0.01 – 2, such as 0.02 – 1 or more preferable 0.05 – 0.5.

7. A process according to any of the preceding claims, wherein the reactor feed is converted in one or more MTA reactors over a bifunctional catalyst comprising a zeolite and a dehydrogenation function.

5 8. A process according to any of the preceding claims, wherein the reactor feed is converted in one or more MTA reactors over a bifunctional catalyst comprising zeolite ZSM-5 and 0.2 – 15 wt% Zn, such as 3 – 15 wt% Zn or 5-15 wt% Zn.

10 9. A process according to any of the preceding claims, wherein the reactor feed is converted in one or more MTA reactors over a bifunctional catalyst comprising zeolite ZSM-5, Zn and 0 – 10 wt% P, such as 0.1 – 8 wt% P or 0.5 – 5 wt% P.

15 10. A process according to any of the preceding claims, wherein the reactor feed is converted in one or more MTA reactors over a bifunctional catalyst comprising zeolite ZSM-5 and Zn, where Al₂O₃ is used as binder to shape the catalyst.

20 11. A plant comprising a first feed stream, a reactor, a separator for separating a reaction effluent into at least an aqueous condensate stream, a liquid hydrocarbon stream and a gas stream,
- means for recycling a first recycle stream from the gas stream to the reactor,
- means for obtaining a first side stream from the gas stream,
- means for removing H₂ removal from the first side stream to obtaining a hydrogen depleted stream, and
- means for adding the hydrogen depleted stream to the reactor,
25 - optionally means for purging at least part of the first recycle stream.

30 12. Plant according to claim 11, wherein the means for removing hydrogen is a membrane, a PrOx reactor, a methanation reactor, a methanol synthesis reactor, oxidation reactor or a catalytic oxidation reactor.

13. An aromatic product obtained by the process according to claim 1 – 10.

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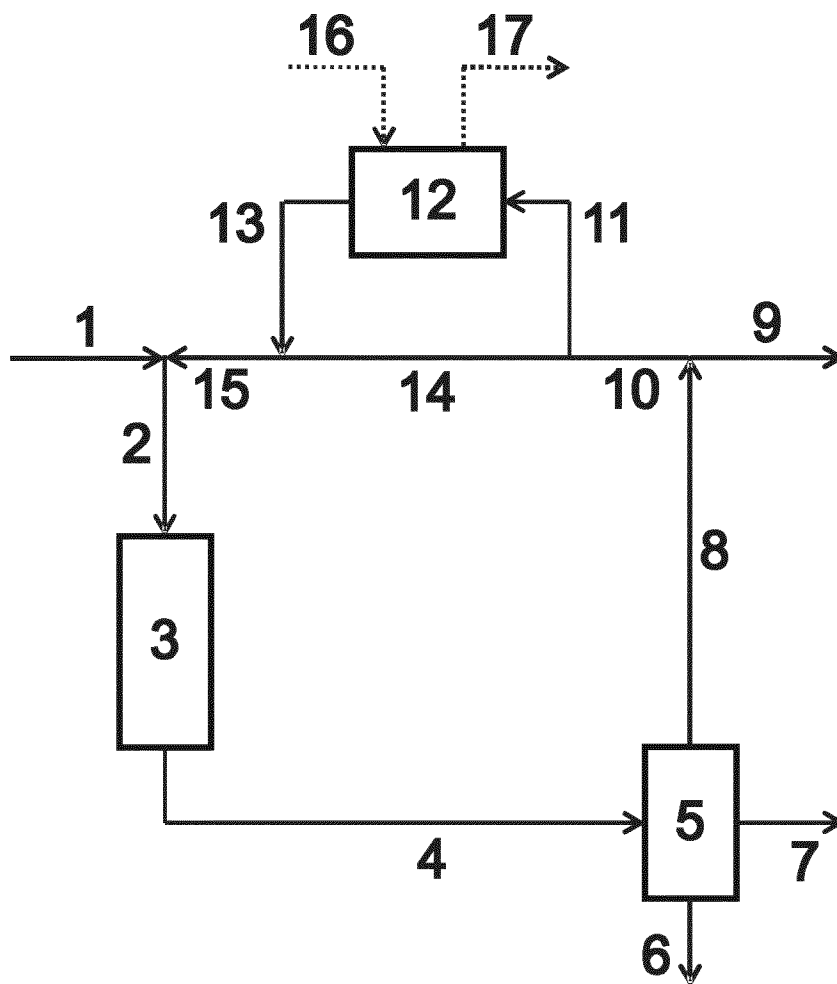


Fig. 1

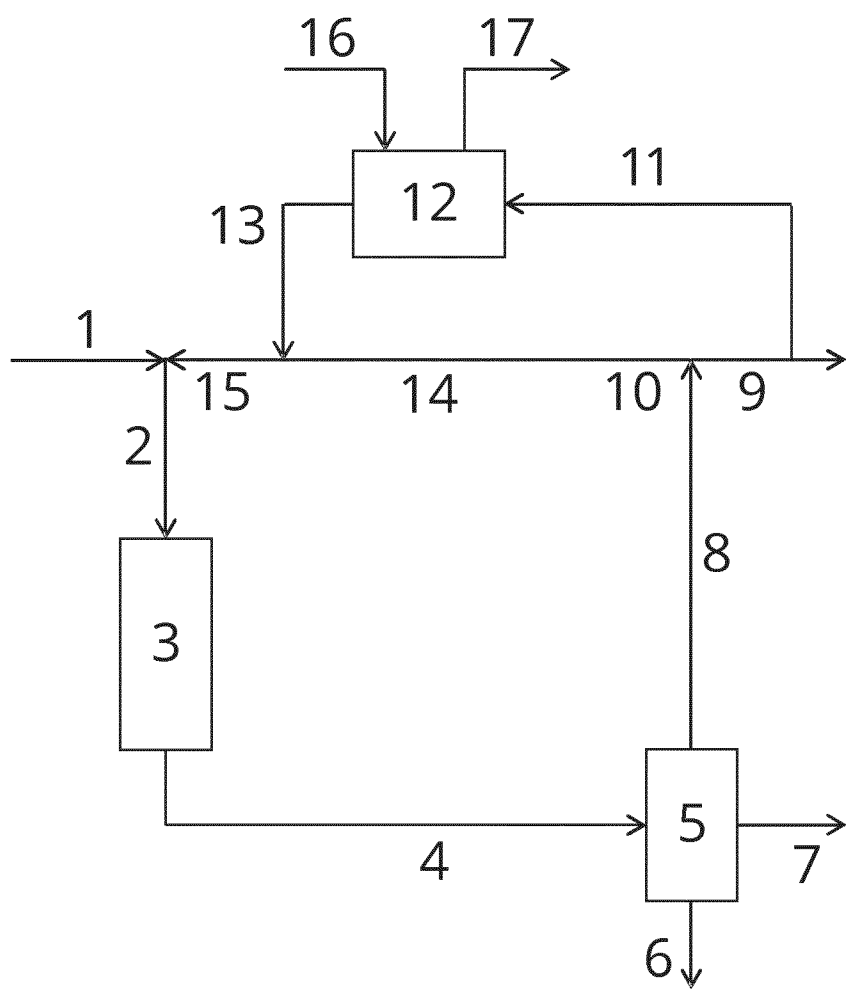


Fig. 2

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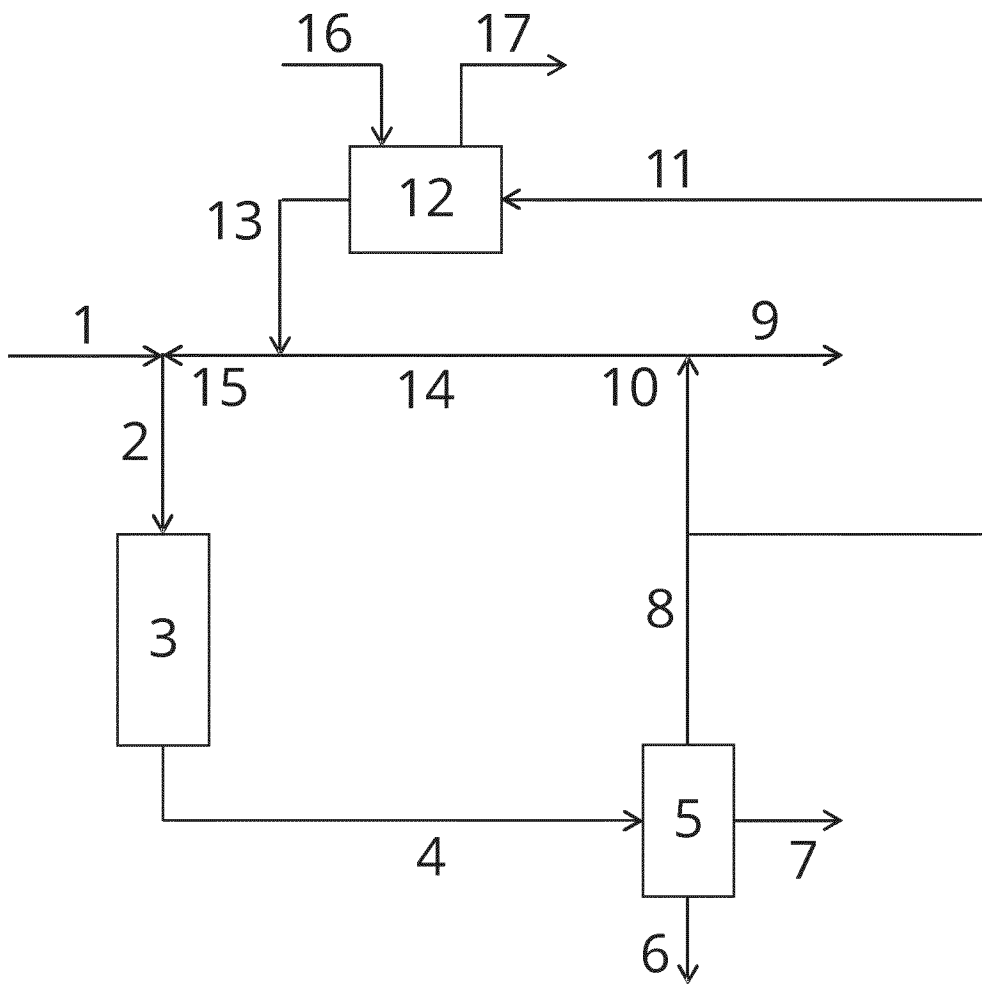


Fig. 3

INTERNATIONAL SEARCH REPORT

International application No

PCT/EP2017/066867

A. CLASSIFICATION OF SUBJECT MATTER
 INV. C10G3/00 C07C1/20
 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)
 C10G C07C

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

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X	EP 3 015 445 A1 (CHINA PETROLEUM & CHEMICAL [CN]; SHANGHAI RES INST PETROCHEMICAL TECHN) 4 May 2016 (2016-05-04)	1-6, 11-13
Y	paragraphs [0002], [0018], [0021], [0029]; figure 1	7-10
A	Sinnott, R. K.: "2. Fundamentals of Material Balances" In: Coulson, J. M., Richardson, J. F.: "Chemical engineering", 1991, XP002774187, ISBN: 0-08-022970-0 vol. 6, page 29, 47, 48, page 48	1-12



Further documents are listed in the continuation of Box C.



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Name and mailing address of the ISA/

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INTERNATIONAL SEARCH REPORT

International application No
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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	paragraphs [0037], [0038], [0067]; figures 1,2	1-12

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A	abstract	1-12
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A,P	paragraphs [0049] - [0051], [0076]; figure 1	1-12

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	paragraphs [0002], [0019], [0024], [0033]	

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No

PCT/EP2017/066867

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