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(54) **PRODUCTION METHOD FOR A MODIFYING COKING ADDITIVE BY DELAYED COKING OF RESIDUE OIL**

(57) Methods for producing oil coke, using delayed coking, are described. A primary feed is heated and charged into a remote stripper of a rectification column, where it is mixed with heavy gasoil as a recirculant and where a secondary feed is produced, which is then heated in a reaction/heating furnace and transferred to a coking chamber, where a coking additive and vapour/liquid coking products are produced. The latter ones are fractionated in the rectification column, forming gas, benzene, light and heavy gasoil and coking bottom products. Calcium oxide/hydroxide is mixed with the secondary feed, and then mixed with heavy gasoil, before being transferred to the coking chamber. Alternatively, calcium oxide/hydroxide is added to the coking bottom products, mixed with heavy gasoil, after which the coking bottom products are either mixed with the secondary feed before feeding it into the coking chamber or they are introduced into the coking chamber directly.

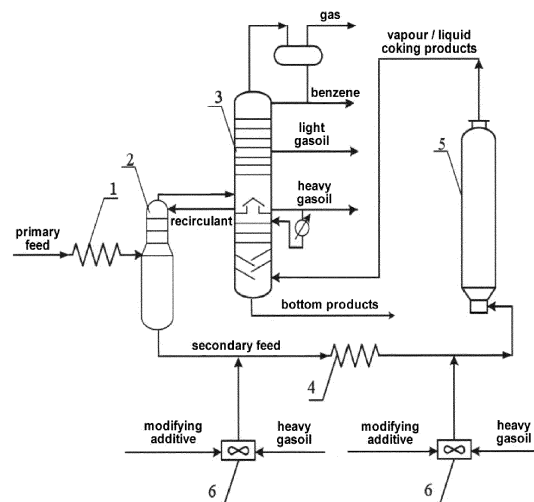


FIGURE 1

Description

FIELD OF THE INVENTION

- 5 **[0001]** The present invention is related to oil processing, in particular to producing oil coke with more than 15% and less than 25% volatiles to be used as a coking additive to coal coking raw materials, using delayed coking.

BACKGROUND OF THE INVENTION

- 10 **[0002]** There exists an oil coke production method, using delayed coking of oil residue, different in that the feed is heated to 300-350°C, charged into the remote stripper of the rectification column, where it is mixed with the recirculant to make a secondary feed; the secondary feed is heated in the reaction/heating furnace to 480-505°C and is charged into the coking chamber to produce coke and vapour/liquid coking products; the latter one are fractionated in the rectification column, producing gas, benzene, and light and heavy gasoil and coking bottom products. Coking bottom products are used as the recirculant (Russian Federation Patent 2209826, cl. C10B 55/00, published in 2003).

- 15 **[0003]** The drawback of that method is in that the target product has a low concentration of volatile products and a high concentration of sulphur, which makes it unsuitable as a coking additive to coal coking feed in blast furnace coke production.

- 20 **[0004]** Closest to both versions of this invention is the method of producing a coking additive by delayed coking of residue oil, which includes charging the feed into the coking chamber at 450-470°C, coking it for 14-24 hours, with recirculation coefficient no higher than 1.2, producing a coking additive and vapour/liquid coking products, fractionating the latter one ones in a rectification column, where gas, benzene and light and heavy gas oil and coking bottom products are generated. As in the similar patent described above, the original coking feed is heated to 300-350°C, charged into the remote stripper of the rectification column, where it is mixed with the recirculant, producing secondary feed (RF Patent 2400518, cl. C10B 55/00, published in 2010).

- 25 **[0005]** The drawback of this method is in that the coking additive produced by coking high-sulphur oil produces a coking additive with high organic sulphur concentration. This kind of additive, being mixed with metallurgical coke, has a negative effect on quality of cast iron and of steel produced by conversion of sulphurous cast irons (steel becomes red short).

- 30 **[0006]** To compensate for the negative effect of organic sulphur in ore and in metallurgical coke used as fuel or reduction agent in blast furnaces, fluxes are contained in the feed, including calcium oxide in the form of lime. The calcium oxides react with the sulphur in the metallurgical coke and convert it into a non-organic form and removed with slags:

- 35
$$\text{CaO} + \text{S} + \text{C} \rightarrow \text{CaS} \downarrow + \text{CO} \uparrow$$

[0007] The higher the sulphur content of the coke, the larger quantity of flux must be added in the form of the calcium oxide into flux. A large quantity of flux in cast iron production overloads the blast furnace, cutting down the output of cast iron.

- 40 **[0008]** There are published data on laboratory tests under stationary conditions, similar to coking in cubes, on effect of alkaline additives on sulphurous raw materials for coking. Much of organic sulphur in the raw materials reacts with potassium hydroxide, converting it into a water-soluble salt, which reduces the sulphur content (N. S. Kazanskaya, E. P. Smidovich and E. P. Sarkisyants "Properties of Oil Cokes, Produced in the Presence of Hydrate of Potassium Oxide"; Izvestiya VUZov "Neft i Gas" (Oil and Gas), 1974, No. 6: 55-58). A considerable quantity of Potassium Hydroxide was introduced, which means that the coke produced had to be washed with water to remove the excess of alkali.

SUMMARY OF THE INVENTION

- 50 **[0009]** The present invention aims at producing a coke additive with a low content of organic sulphur compounds, using delayed coking.

- [0010]** Methods for producing oil coke, using delayed coking, with the volatile components making up more than 15%, but less than 25%, to be used as a coking additive into feed for oil coking, are provided. The primary feed may be heated and charged into a remote stripper of the rectification column, where it may be mixed with heavy gasoil as a recirculant and where the secondary feed may be produced, which may then be heated in the reaction/heating furnace and transferred to the coking chamber, where a coking additive and vapour/liquid coking products may be produced. The latter ones may be fractionated in the rectification column, forming gas, benzene, light and heavy gasoil and coking bottom products. Calcium oxide or hydroxide may be mixed with the secondary feed as a modifying additive, mixed with heavy gasoil at a ratio of 25-35:65-75, before it may be transferred to the coking chamber. Alternatively, calcium oxide or hydroxide may

be added to the coking bottom products as a modifying additive, mixed with heavy gasoil as a modifying additive in a ratio of 25-35:65-75, after which the coking bottom products may be either mixed with the secondary feed before feeding it into the coking chamber or they may be introduced into the coking chamber directly, and the modifying additive introduced into the coking chamber may amount to 0.5-10.0 mass % of the primary feed.

[0011] In the first version of this method, the goal may be achieved by producing a coking additive with delayed coking of residue oil, using the following sequence of operations: the feed may be heated, charged into a remote stripper of the rectification column to have it mixed with heavy gasoil, used as a recirculant, producing a secondary feed, heating the secondary feed in the reaction-heating furnace and feeding it into the coking chamber, where a coking additive and vapour/liquid products form, and fractioning the latter one in a rectification column, producing gas, benzene, light and heavy gasoil and coking bottom products. According to one aspect of the present invention, prior to introducing the secondary feed into the coking chamber, it may be mixed with a modifying additive: calcium oxide (CaO) and/or calcium hydroxide (Ca(OH)₂) either before heating it in the reaction/heating furnace or after that. The quantity of the modifying additive introduced into the coking chamber may comprise 0.5-10.0% (mass) of the primary feed, while the modifying additive may be mixed, a-priori, with heavy gasoil at a 25-35:65-75 ratio. The modifying additive may be mixed with the secondary feed before and/or after the reaction/heating furnace.

[0012] In the second version of the method, the goal may be achieved of producing a coking additive by delayed coking of residue oil, including heating the primary feed, feeding it into a remote stripper of the rectification column to mix it with heavy gasoil used as a recirculant and producing a secondary feed, heating the secondary feed in the reaction/heating furnace and transferring it into the coking chamber, where a coking additive and vapour/liquid coking products form, and fractioning the latter one in a rectification column, where gas, benzene light and heavy gasoil and coking bottom products form. In accordance with this method, calcium oxide and/or calcium hydroxide as a modifying additive, mixed, a-priori, with heavy gasoil at a 25-35:65-75 ratio, then the coking bottom products are either mixed with the secondary feed before charging it into the coking chamber or introduced into the coking chamber directly, with the modifying additive comprising 0.5-10.0% (mass) of the primary feed.

[0013] In both these versions, the secondary feed may be heated in the reaction/heating furnace at 450-490°C.

[0014] In both versions of the methods, the quality of heavy coking gasoil, removed from the rectification column and coking bottom products, may be controlled by circulating irrigation of the bottom part of the column with heavy gasoil, and by varying the quantity of irrigation gasoil onto the plates of the column, the quality and quantity of heavy gasoil and coking bottom products may be controlled.

[0015] In both versions, it may be feasible to spray an anti-foaming dope into the upper part of the coking chamber to reduce foaming during the coking in the presence of calcium oxide and/or hydroxide.

[0016] Introducing the modifying additive into the coking materials with the secondary feed and/or coking bottom products may ensure that the quantity of the organic forms of sulphur in the produced modified coking additive may be reduced due to the fact that organic sulphur compounds contained in the primary feed react with calcium oxide and/or hydroxide, forming a non-organic, non-active sulphur compound. Adding less than 0.5% mass of calcium oxide and/or hydroxide in relation to the mass of the primary feed may not be feasible because this would not bring down the sulphur content in the coking additive considerably, while adding more than 10% of metal oxide with respect to the primary feed may be unfeasible because this would increase ash content in the coking additive produced.

[0017] Circulating irrigation at the bottom of the rectification column, which allows the control of the quality of final heavy gasoil and coking bottom products, in particular the start and finish temperatures of their boiling, and consequently control their viscosity, may ensure that the calcium oxide and/or hydroxide suspension in heavy gasoil has the right concentration for pumping it over. Thus, if heavy gasoil let out of the column has a high boiling temperature, then its viscosity is also high, which may make pumping difficult when the calcium concentration in the suspension exceeds 35%. Increasing circulating irrigation at the bottom of the column may ensure condensation of high-boiling-temperature components of heavy gasoil and their transition into the coking bottom products product. This may reduce viscosity of both the heavy gasoil and coking bottom products.

[0018] Introducing an anti-foaming dope into the upper part of the coking column may prevent coke foams getting into the rectification column and its becoming coked up due to intensive foaming in the process of coking of petroleum raw materials in the presence of calcium oxide and/or hydroxide. In those cases when the feed is mixed with calcium oxide, foaming does not happen during the heating of the modifying suspension in heavy gasoil up to 300°C, while, when the feed is mixed with calcium hydroxide, foaming occurs even when the temperature only exceeds 80°C.

BRIEF DESCRIPTION OF THE DRAWINGS

[0019]

Figure 1 shows a schematic diagram of an illustrative system for carrying out a method in accordance with version 1 of the present invention; and

Figure 2 shows a schematic diagram of an illustrative system for carrying out a method in accordance with version 2 of the present invention.

DETAILED DESCRIPTION OF THE INVENTION

[0020] The methods of the present invention - in either the first or second version - generally operate as follows.

[0021] The input (primary) sulphurous feed may be heated in the furnace 1 to 250-390°C and introduced into the remote stripper 2 of the rectification column 3, where the recirculant may also be introduced: heavy coking gasoil from the rectification column. The secondary feed, formed by mixing the primary feed with the recirculant, may be heated in the reaction/heating furnace 4 up to 450-490°C and introduced into the coking chamber 5, where a coking additive and vapour/liquid coking products form, and transferred - via a slam pipe into the rectification column, where they are fractionated, producing gas, benzene, light and heavy gasoil and coking bottom products.

[0022] In version 1, prior to charging the secondary feed into furnace 5, it may be mixed with the modifying additive - calcium oxides and/or hydroxides - before or after the reaction/heating furnace 4, and the modifying additive had been a-priori mixed with heavy gasoil in a 25-35:65-75 ratio, forming a suspension of the modifying additive in heavy gasoil, using for this mixer 6 or a disintegrator.

[0023] In accordance with the second version of the proposed method, calcium oxide and/or hydroxide may be introduced into the coking bottom products - as a modifying additive, mixed a-priori with heavy gasoil in a ratio 25-35:65-75, forming a suspension of the modifying additive in heavy gasoil, using the mixer 6 or a disintegrator for example. Then the coking bottom products may either be mixed with the secondary feed prior to its being charged into the coking chamber or may be charged into the coking chamber directly.

[0024] In both cases, calcium oxide or hydroxide concentration may amount to 0.5-10.0% (mass) of the primary feed.

[0025] In both versions, foaming may be prevented by introducing a mixture of the modifying additive with heavy gasoil at the outlets of the pumps (not shown in the Figures) that pump the coking bottom products and secondary feed over to the coking chamber.

[0026] In addition, both versions of this method may provide for circulating irrigation with heavy gasoil, and the quantity and quality of the heavy gasoil and coking bottom products let out of the rectification column may be controlled by varying the quantity of circulating irrigation supplied to the first plate underneath the accumulator of the rectification column.

[0027] Also, in both versions, an anti-foaming additive may be sprayed into the upper section of the coking chamber (not shown).

[0028] The following examples illustrate the applications of each version of this method.

Example 1 (Version 1)

[0029] The primary feed (tar made up of a mixture of West-Siberian and Arlan oils), density 1.025 g/cm³, Conradson coking capacity of 24 maa %, sulphur content 3.21% was coked in a commercial delayed coking unit as follows:

[0030] The primary feed was heated in a tubular furnace at 310 °C, then charged into a remote stripper of the rectification column, where it was mixed with heavy coking gasoil as a recirculant, 10% of the primary feed. The secondary feed was mixed with calcium oxide, mixed a-priori with heavy gasoil at a 25:75 ratio, forming a calcium oxide suspension in heavy gasoil. Calcium oxide amounted to 2.5 mass % over the primary feed, then it was heated in the furnace at 470 °C and charged into the coking chamber, where a coking additive and vapour/liquid coking products formed. The latter ones were charged, via a slam pipe, to the lower part of the rectification column, where they were fractionated, forming gas, benzene, light and heavy gasoil and coking bottom products.

[0031] The coking conditions, material balance and quality of the coking additive produced in Example 1 are shown in the Table.

[0032] Version 1 was implemented in Examples 2-5, and the coking conditions and results of coking are shown in the Table.

Example 6 (Version 2).

[0033] The same primary feed as in Example 1 was heated in a tubular furnace at 310 °C, then charged into a remote stripper of the rectification column, where it was mixed with heavy coking gasoil as a recirculant, 10% of the mass of the primary feed. The secondary feed produced was heated in the furnace at 485 °C and transferred to the coking chamber, where a coking additive and vapour/liquid coking products formed. The latter ones were, via a slam pipe, charged into the bottom part of the rectification column, where they were fractionated, forming gas, benzene, light and heavy gasoil and coking bottom products. The produced coking bottom products were mixed with calcium oxide, a-priori mixed with heavy coking gasoil at 30:70, forming a calcium oxide suspension in heavy gasoil, and charged into the coking chamber directly. Calcium oxide concentration was 5 mass % of the primary feed.

[0034] Using Version 2, two more examples of coking are shown in Examples 7 and 8. The input data of that process and the quality of the coking additives in Examples 6-8 are also shown in the Table.

[0035] To compare the two versions of the method proposed, a coking additive was produced, using the prototype method.

Example 9 (using the prototype method)

[0036] The same feed as in Example 1 was heated in the furnace up to 320 °C, then transferred to the remote stripper of the rectification column, where it mixed with heavy coked gasoil as a recirculant, 10% of the primary feed. This secondary feed was heated at 470 °C in the furnace and transferred into the coking chamber, where a coking additive and vapour/liquid coking products formed. The latter ones were charged, via the slam pipe, into the lower section of the rectification chamber, where they were fractionated, forming gas, benzene, light and heavy gasoil and coking bottom products. Data related to Example 9 are shown in the Table.

[0037] As can be seen from the data presented, coking with a modifying additive reduces sulphur content in the coking additive produced (down to 0.65-2.87%, while its content in the prototype was 3.62%). This occurs because some of the sulphur binds with calcium, forming calcium sulphide (CaS). The greater quantity of the modifying additive mixed with the primary coking feed, the greater the decrease of organic sulphur concentration in the coking additive, however ash content increases in the coking additive at the same time. Ash exists in the form of calcium sulphide, which in future, getting into the blast furnace as a part of metallurgical coke in the form of fuel or a reducing agent, incorporates with slag, cutting down the required quantity of fluxes introduced for desulphurisation of sulphur contained in the ore and fuel, or: in the form of calcium oxide and/or hydroxide - the main flux components - which have not reacted with sulphur, which also decreases the quantity of required fluxes as well as supporting further decrease of sulphur in cast irons.

[0038] The modifying additive is introduced into the secondary feed or coking bottom products gasoil in the form of calcium oxide and/or hydroxide suspension in heavy coking gasoil. When the modifying additive is introduced at the outlet of the reaction/heating furnace, temperature at the inlet of the coking chamber goes down, and for this reason temperature of the secondary feed at the furnace outlet can be increased to 490 °C.

[0039] When the calcium hydroxide suspension in heavy gasoil is prepared at temperatures in excess of 80 °C, it is accompanied by intensive foaming. It is therefore recommended to prepare that suspension at low (below 80 °C) temperatures. Also, when using calcium hydroxide as a modifying additive, which requires a large quantity of calcium (10% of the primary feed) and to avoid having to considerably raise the temperature of the secondary feed at the furnace outlet - if the modifying additive is introduced after the furnace - the suspension needs to have the highest possible concentration of calcium. However this would make the suspension more viscous, which would cause difficulties with pumping. In this case it is necessary to have gasoil used for making the suspension as viscous as possible. To control viscosity of heavy gasoil so as to ensure that the suspension has viscosity required for normal pump operation, circular irrigation is applied to the plates of the lower part of the rectification column. Supplying greater quantities of cooled heavy gasoil, used for circulating irrigation onto the first plate under the accumulator of the rectification column, promotes condensation of high-boiling-temperature (and consequently, high viscosity) fractions of heavy gasoil and their transition to the coking bottom products, which pushes down the end of boiling temperature of heavy gasoil, reduces its viscosity and, at the same time, reduces the start of boiling temperature of coking bottom products and their viscosity.

[0040] To reduce foaming and avoid completely the transfer of coking foam to the rectification column in the process of coking in accordance with Examples 2 and 5-8, an anti-foaming dope was added in the top part of the coking chamber.

[0041] When the modifying additive was introduced in the form of a suspension in heavy coking gasoil mixed with coking bottom products into a coking chamber directly, simultaneously with decrease of the organic sulphur content in the coking additive, the output of the latter increases. This happens because the coking bottom products consist of the least volatile fractions formed during coking, characterised by high density and coking capacity; using coking bottom products in the coking process boosts up the output of the coking additive.

[0042] It follows that use of one of the versions of the method proposed produces a coking additive with a lower content of organic forms of sulphur than with the prototype method.

[0043] Moreover, including coking bottom products as a part of the secondary feed increases the output of the coking additive.

TABLE - INPUT COKING DATA, MATERIAL BALANCE AND TARGET PRODUCT QUALITY

	Version 1 Method					Version 2 Method			Prototype
	1	2	3	4	5	6	7	8	
Examples									9
Characteristics									
1. Modifying additive	CaO	CaO	CaO	Ca(OH) ₂	Ca(OH) ₂	CaO	Ca(OH) ₂	Ca(OH) ₂	-
2. Quantity of modifying additive, feed %	2.5	10.0	5.0	5.0	2.5	5.0	5.0	10.0	-
3. Modifying additive concentration in heavy gasoil, %	25.0	25.0	25.0	25.0	35.0	30	30	25	-
4. Modifying additive introduced location	Before furnace	Before furnace	After furnace	Before furnace	After furnace	Coking chamber	Secondary feed	Coking chamber	-
5. Temperature of secondary feed at furnace outlet	470	470	485	470	485	485	485	480	470
6. Circulating watering quantity applied	6.0	6.0	6.0	6.0	8.5	8.0	8.0	6.0	6.0
7. Anti-foaming dope (yes, no)	No	Yes	No	No	Yes	Yes	Yes	Yes	Yes
8. Foam height over coke, m	5.0	3.0	4.0	5.0	3.0	5.0	5.0	5.0	4.0
9. Material balance of the process, mass %:									
- gas (in respect to C ₄)									
- benzene (C ₅ - 180 °C) fraction	11.8	12.7	12.0	9.6	8.2	10.5	8.5	9.3	13.4
- light gasoil (180-350 °C)	6.07	6.20	6.1	4.8	4.9	6.1	5.1	4.4	6.03
- heavy gasoil (350-500 °C fraction)	26.5	21.0	24.2	32.1	36.2	24.2	29.6	25.3	25.27
- bottom products (>500 °C fraction)	17.03	19.1	18.4	12.0	10.7	15.4	14.0	12.0	18.6
- coke	5.0	5.0	5.0	5.0	5.0	8.0	5.0	6.0	5.0
	33.6	36.0	34.3	36.5	35.0	35.8	37.8	42.0	31.7
10. Quality of coking additive, %:									
- sulfur content	2.87	1.53	1.80	1.54	2.11	1.75	1.61	0.65	3.62
- ash content	10.15	30.7	24.15	15.84	12.96	25.1	16.3	31.1	0.24
- volatiles content	16.7	17.3	18.1	17.8	19.0	19.0	17.9	19.4	16.8

Claims

1. A method for producing a modified coking additive with delayed coking of residue oil, comprising the steps of heating and charging a primary feed into a remote stripper of a rectification column, wherein it is mixed with heavy gasoil as a recirculant, and wherein a secondary feed is produced, wherein the secondary feed is heated in a reaction/heating furnace and transferred to a coking chamber, wherein a coking additive and vapour/liquid coking products are produced, wherein the latter ones are fractionated in the rectification column, producing gas, benzene, light and heavy gasoil and coking bottom products, wherein the secondary feed, before being transferred to the coking chamber, is mixed with calcium oxide or hydroxide as a modifying additive, and mixed with heavy gasoil at a 25-35:65-75 ratio, and wherein the modifying additive is charged into the coking chamber and forms 0.5-10.0 mass % of the primary feed.
2. The method according to claim 1, wherein the secondary feed is mixed with the modifying additive either before it is transferred to the reaction/heating furnace or after it.
3. The method according to claim 1, wherein the secondary feed is heated in the reaction/heating furnace at 450-490 °C.
4. The method according to claim 1, further comprising the step of providing circulating irrigation with heavy gasoil of a lower part of the rectification column, wherein a quality and a quantity of heavy gasoil and coking bottom products obtained from the rectification column are controlled by varying a quantity of circulating irrigation applied to a plate of the column.
5. The method according to claim 1, further comprising the step of spraying an upper part of the coking chamber with an anti-foaming additive.
6. A method of producing a modifying coking additive with delayed coking of petroleum residues, comprising the steps of heating and placing a primary feed into a remote stripper of a rectification column, wherein it is mixed with heavy gasoil as a recirculant and wherein a secondary feed is produced, which is then heated in a reaction/heating furnace and transferred to a coking chamber, wherein a coking additive and vapour/liquid coking products are produced, wherein the latter one ones are fractionated in the rectification column and form gas, benzene, light and heavy gasoil and coking bottom products, wherein calcium oxide or hydroxide are added to the coking bottom products as a modifying additive, mixed with heavy gasoil at a ratio of 25-35:65-75, after which the coking bottom products are either mixed with a secondary feed before feeding it into the coking chamber or it is introduced into the coking chamber directly, and wherein the modifying additive introduced into the coking chamber amounts to 0.5-10.0 mass % of the primary feed.
7. The method according to claim 6, wherein the secondary feed is heated in the reaction/heating furnace at 450-490 °C.
8. The method according to claim 6, further comprising the step of providing circulating irrigation with heavy gasoil of a lower part of a rectification column, wherein a quality and a quantity of heavy gasoil and coking bottom products obtained from the rectification column are controlled by varying a quantity of circulating irrigation applied to a plate of the column.
9. The method according to claim 6, further comprising the step of providing circulatory irrigation of heavy gasoil in a lower part of the rectification column, wherein a quality and a quantity of heavy gasoil and coking bottom products, let out of the rectification column, are controlled by varying a quantity of circulating irrigation applied to a plate of the column.

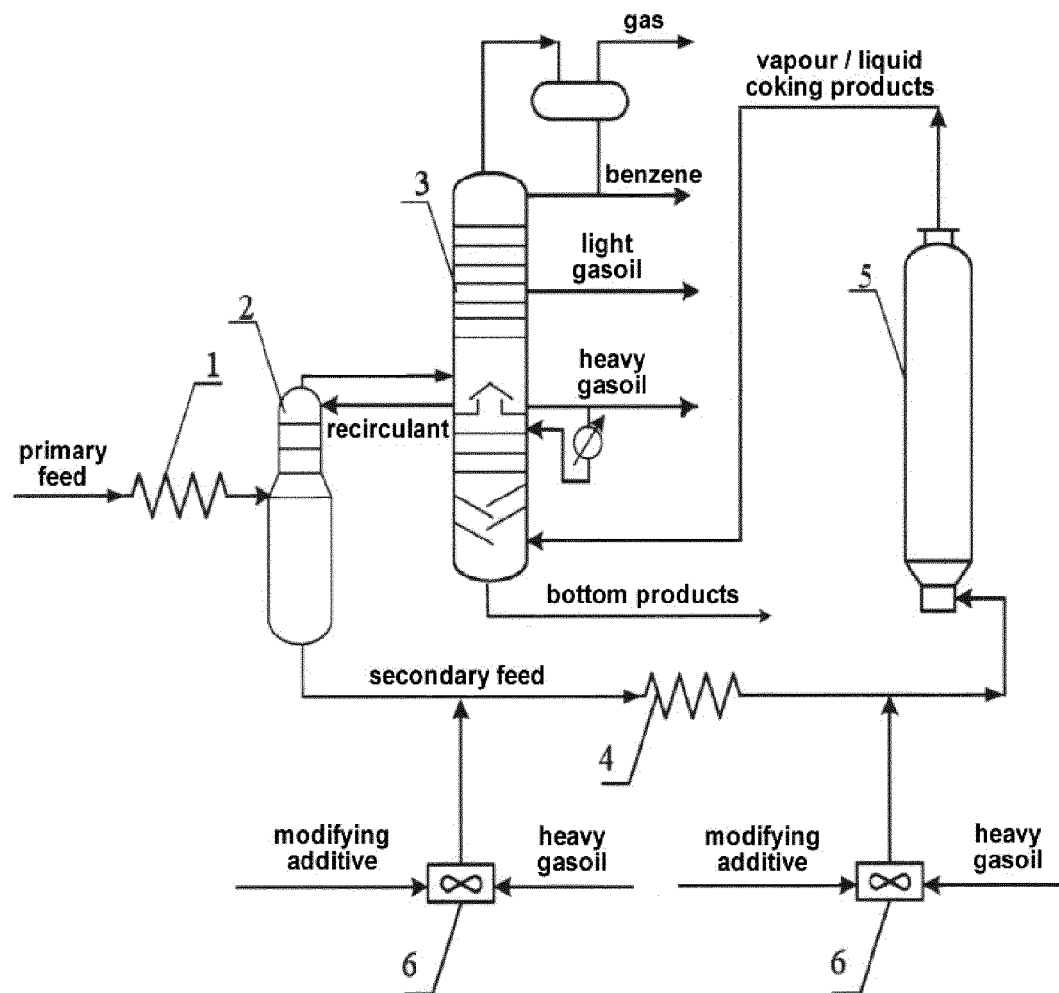


FIGURE 1

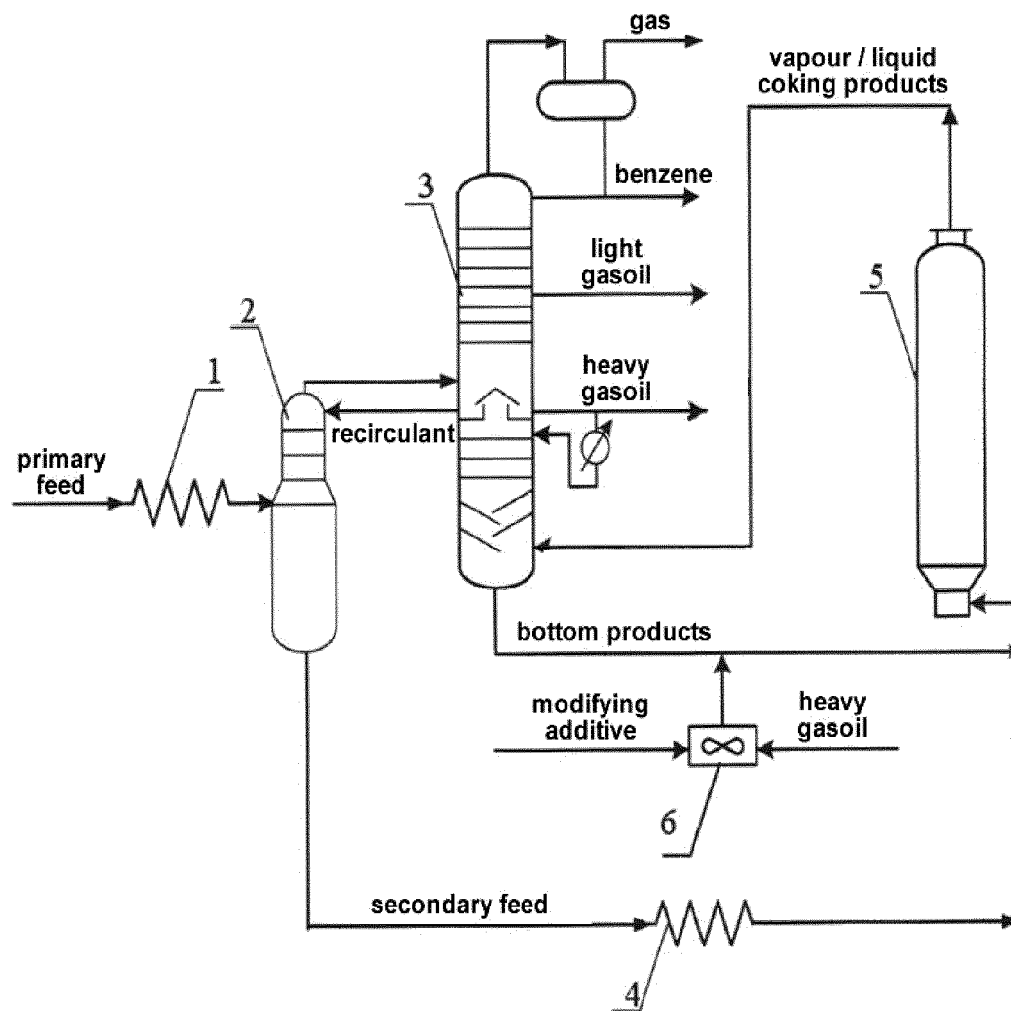


FIGURE 2



EUROPEAN SEARCH REPORT

Application Number
EP 15 16 3072

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DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X	RU 2 495 078 C2 (000 PROMINTEKH [RU]) 10 October 2013 (2013-10-10) * figures 1,2 * * table 1 * * claims 1-9 *	1-9	INV. C10B55/00 C10B57/06
A	RU 2 469 068 C1 (GUP INST NEFTEKHIMPERERABOTKI RESPUB BASHKORTOSTAN GUP INKHP RB [RU]) 10 December 2012 (2012-12-10) * claims 1-4 *	1-9	
A	WO 2012/158064 A1 (OBSHESTVO S OGRANICHENNOI OTVESTVENNOSTYU PROMINTEKH [RU]) 22 November 2012 (2012-11-22) * figure 1 * * claims 1-4 *	1-9	
X,P	WO 2015/038023 A1 (OBSHHESTVO S OGRANICHENNOI OTVETSTVENNOST YU PROMINTEKH [RU]) 19 March 2015 (2015-03-19) * figures 1,2 * * claims 1-9 *	1-9	TECHNICAL FIELDS SEARCHED (IPC) C10B C10G
The present search report has been drawn up for all claims			
Place of search The Hague		Date of completion of the search 27 August 2015	Examiner Zuurdeeg, Boudewijn
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

EPO FORM 1503 03.82 (P04C01)

**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 15 16 3072

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
The members are as contained in the European Patent Office EDP file on
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27-08-2015

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
RU 2495078 C2	10-10-2013	EA 201201037 A1 RU 2011142086 A	30-04-2013 27-04-2013
RU 2469068 C1	10-12-2012	-----	-----
WO 2012158064 A1	22-11-2012	EA 201200588 A2 WO 2012158064 A1	28-12-2012 22-11-2012
WO 2015038023 A1	19-03-2015	NONE	-----

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- RU 2209826 [0002]
- WO 2400518 A [0004]

Non-patent literature cited in the description

- **N. S. KAZANSKAYA ; E. P. SMIDOVICH ; E. P. SARKISYANTS.** Properties of Oil Cokes, Produced in the Presence of Hydrate of Potassium Oxide. *Izvestiya VUZov "Neft i Gas" (Oil and Gas)*, 1974, 55-58 [0008]