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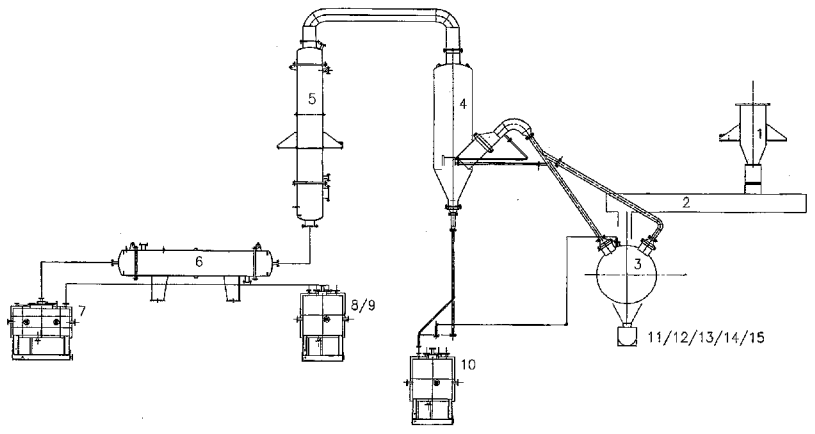
(54) Title: DEPOLYMERIZATION PROCESS FOR PRODUCING OFF-SPEC CRUDE AND CARBON BLACK FROM SCRAP TIRES



(57) Abstract: The present invention provides an economical process for the producing of off-spec crudes and carbon black from vulcanized rubber chips, derived from scrap tires. The process comprises the de-polymerization of rubber chips by breaking down the number of carbon atoms in molecules. The de-polymerization process utilizes endothermic heat to decompose the long chain polymers of rubber chip into short petroleum hydrocarbon. The vulcanized rubber chip is de-polymerized into an off-spec crudes hydrocarbon vapor and solid residue.

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DEPOLYMERIZATION PROCESS FOR PRODUCING OFF-SPEC CRUDE AND CARBON BLACK FROM SCRAP TIRES

FIELD OF THE INVENTION

This invention relates to processes for the recovery of off-spec crudes and solid residue "carbon black" from vulcanized rubber chips.

BACKGROUND OF THE INVENTION

The years between 2003 and 2011 were particularly difficult years to evaluate the oil economy. Annual global crude oil discovery rates peaked in the 1960s and despite huge advances in exploration technology, the source of supply is declining. The major challenge in finding a replacement for crude oil is to identify a source that will be replenished. There is a dire need of oil to generate industries for heating while there is a shortage of crude oil from natural sources.

Two major sources deem to be replacement of natural explored crude are in one case, oil shale, which is organic material known as kerogen is known to be impractical. It takes more energy to produce oil from shale than the energy to produce oil, with Energy return on investment (EROI) of less than 1.

Another source of oil replacement is in Ethanol and biodiesel, which are crop-based fuels, produced from corn and soya beans respectively. Crop based fuels faces great challenges as a replacement for oil as these are number one source of food calories. There is no easy solution to replacing oil as a heating or as a liquid transportation fuel. Meanwhile, there is an ever increasing global problem of the disposal of discarded waste tires, resulting in the millions of metric tonnes being stockpiled or discarded indiscriminately. There is national concern to health management as the costs of disposal of solid waste are massive waste of resources. However, this source

of solid organic waste product, namely vulcanized scrap tires that is being replenished daily and globally could be depolymerized into off-spec crudes, suitable for heating and driving static equipment as pumps and system via an off-spec crudes generator. The idea of recovering off-spec crudes and solid residue "carbon black" from rubber chips wastes of scrap tires is old in the art. However, processes need to be developed which utilize both a continuous system that can break down the long chain of polymer atom molecules into short chain of petrol hydrocarbon with a minimal energy input, with an EROI 1: 7.

Incineration or heat based via direct furnace-burner of the organic portion of the rubber chips does not appear to be the solution. Off gases produced during intense heat incineration contain air pollutants such as sulfur dioxide (SO2), Nitrogen oxide ($NO_{,,}$), carbon monoxide (CO) and ash. To avoid air pollution, these pollutants must be diminished or trapped in the process. This requires costly devices such as electro-static precipitators and the like. What is needed is an efficient, economical method for handling rubber chip wastes produced by the society from the millions of discarded waste scrap tires, which can recover off- spec crudes and residue solid "carbon black" from the organic portion while substantially reducing the volume of gaseous discharge effluents, which must be treated to eliminate air pollution during processing.

Processes currently used in reclaiming off-spec crudes and carbon black; generally begin with pyrolysis of such waste material. In the current pyrolysis processes, the problem is the production of solids within the waste products. During pyrolysis, an inert carrier gas is used and fine particulate are unavoidably passed through the conventional solid-gas scrubbing units. The solids arise from the quick volatizing of material and solid particulates form from the vapor stage by distillation of the vapors. Solids entrained in the pyrolysis vapors must be stopped as the particulate in pyrolytic oil can cause abrasion of piping lines and can plug openings or other components within a solid or solid transfer system. Furthermore, the ash and particulate emission will increased when the pyrolytic oil is burned for consumption.

Prior method of gravitational filtration or centrifuging has not been successful in resolving the solid removal issues. Therefore, an improvement in the solids separation while retaining the inherent fluidity of the off-spec crudes is the desire recourse.

In this invention, the desired result is being achieved by utilizing a unique clarification process at 80 degree Celsius with a centrifugation-purification process. Together with a reflux light fraction into condenser No.1, a quality reflux off-spec crudes light fraction can be achieved economically; as the said dual proprietary catalyst heating system (manufactured by Energeia [S] Pte. Ltd) and centrifuging-purifying components (manufactured by Alfa Laval Ltd) ensure a low consumption of within 270 amperes of 80-400 degree Celsius.

SUMMARY OF THE INVENTION

According to the present invention there is provided a process and apparatus related to the de-polymerization of organic vulcanized rubber chips for the production of valued off-spec crudes of heavy and light fractions and subsequent separation of solid residue "carbon black" for recycle back into the economy. Organic fractions of a mixture of vulcanized rubber chips and crumbs of a size of 10-50 millimeter (mm) are subjected to a de-polymerization process. The de-polymerization consists of an operation wherein the dried organic fraction mixture is combined with an endothermic source of heat and a carrier gas which is non-reactive with respect to the product of de-polymerization under regulated turbulent push-throw conditions of the mixture and particulate matter in a de-polymerized zone maintained at a temperature of about 300 degree Celsius to about 450 degree Celsius. Hydrocarbon residue solid "carbon black" and condensable off-specs crudes are formed as the de-polymerization products. The off-spec crudes vapors produced in the de-polymerized process contain solids arises from inorganic particulates formed from the decomposition of vapors. Heavy fraction off-spec crudes are produced by quenching at least a portion of the de-polymerized

vapor. Solid particulate becomes entrained in these heavy off-spec crudes. During the continuous rapid condensation at the condenser No. 2 at temperature of 80 degree Celsius, medium heavy fraction off-spec crudes with lesser substantiated particulate matters is produced. The medium heavy fraction is further condensed at the cooler condenser at a temperature of 80 degree Celsius. The medium heavy fraction is generally made up of American Petroleum Institute (API) gravity < 29°, heavy off-spec crudes which is further clarified, centrifuged and purified to a light fraction. The light fraction produced at a temperature 80 degree Celsius upon clarification-centrifugation-purification contains a substantially reduced amount of particulate matters. The solid residue separated from the distillation of the de-polymerized vapor can be reclaimed as "carbon black".

BRIEF DESCRIPTION OF THE DRAWING

The accompanying drawing illustrates a schematic outline and arrangement of equipment for carrying out the process of this invention.

DETAILED DESCRIPTION OF THE INVENTION

An economical process for the recovery of off spec crude and carbon black from vulcanized rubber chips, derived from scrap tires. The process comprises the de-polymerization of vulcanized rubber chips by breaking down the number of carbon atoms in molecules to a range of 20-27 at standard atmospheric pressure at an approximate boiling temperature of 350-450 degree Celsius. The de-polymerization process utilizes endothermic heat to decompose the long chain polymers of rubber chip into short petroleum hydrocarbon with a maximum length of 27 carbons. Crude oil consist of 84-87% carbon atoms, 11-14% hydrogen atoms, 0%-6% sulfur atoms and less than 1% nitrogen atoms, oxygen atoms, metals and salts. This process consist of endothermic heating of rubber chips via a stationary jacketed reactor; that propels the rubber chips forward by extruders arms welded to a heated-up rotating shaft within the reactor. Heating of the rubber chips in the stationary reactor from an endothermic heat source, generated from a sodium nitrates catalyst heating system. The proprietary heating tanks-clarification system is especially designed-manufactured by Energeia (S) Pte. Ltd. (utilized particularly for this de-polymerization process only) together with a proprietary centrifuging system from Alfa Laval Company forms an integral part of this process system. The rubber chip is de-polymerized into an off-spec crudes hydrocarbon vapor and solid residue. The vaporized off-spec crudes with entrained particulate matter is quenched in condenser no.l; off-loading heavy end off-spec crudes fraction of approximate 35 carbon atoms in molecules into an off-spec crudes heavy oil tank; while the middle distillates off-spec crudes fraction hydrocarbon vapor is substantially free of a heavy fraction containing entrained particulate matter, proceed to condenser no.2 and cooled into

off-spec crudes liquid form by a cooler heat exchanger. The depolymerized off-spec crudes hydrocarbon vapor is condensed and cooled into off-spec crude of 20-27 atoms in molecules, which is further centrifuged and purified producing a light fraction containing entrained particulate matter and pumped into an off-spec crude light oil tank, the solid residue "carbon black" is propel forward and exit via a vibrator-chute via an air lock into a series of conveyors with cutting blades that disintegrate the carbon black into powdery form into a retaining silo with vibrator and an on-off valve for dislodging into packing bags.

The process herein is a distillation process for solid organic waste as in vulcanized rubber chips or carboneous organic waste matters which are produced by the air-land transportation and the motoring communities globally. The solid organic waste is treated and distillate to produce and recover valuable products as in heavy and light fractions of hydrocarbon off-specs oil and solid residue "char" "carbon black". This process hereby said "de-polymerization" is for conversion and/or volatilization of the organic material in the rubber chips to useful organic fuel values such as hydrocarbon off-specs oil and carbon black. The degree of segregation of organic matters from the solid wastes as in rubber chips and/or crumbs can impose uneconomical cost factors on the overall process.

Solid wastes can be segregated by shredding by using conventional equipment and processes. Preferably, the size of the organic solid matter has a maximum dimension of 50 millimeter (mm) x 50 millimeter (mm) and smaller as in 10 millimeter (mm) size of rubber crumbs. By the term "maximum dimension" is meant the largest dimension, either in width or length. The individual size and shape of the organic particles, as well as the density, can affect the heat transfer to the particles. Any changes in size can therefore, necessitate adjustment of residence times within the de-polymerized zone to ensure that the particles of organic matters within the rubber chip are heated to the desired reaction temperature. For this reason, particle size greater than the 50mm do not provide the designated high rate of endothermic heat transfer which is essential to the de-polymerization process.

In the process, de-polymerization is conducted by endothermic heating the organic solid waste aka rubber chips to a temperature between 300 degrees Celsius to about 450 degrees Celsius. To accomplish de-polymerization in the process herein, a preferred heating rate is from 10 degree Celsius/minute to about 20 degree Celsius/minute until the reactor de-polymerization zone reaches a holding temperature of 400 degree Celsius. During de-polymerization, the rubber chips are entrained in a turbulent gaseous stream composed of inert gas, waste solids and hot particulate carbon black. The residence time is 60 minutes per cycle at a temperature of about 400 degree Celsius. The rubber chips are composed of natural and synthetic rubber with carbon black, synthetic fibers and short length of binding steel wires. The relationship between temperature, pressure and residence time can be varied to optimize yields of de-polymerized vapor and residue solid. If the temperature and/or residence times are too low, the vaporization and de-polymerized of solid waste is incomplete. When the temperature and/or residence time is too high, the de-polymerized products are degraded producing low yields of products and related fuel values. By the term "turbulent stream" is meant a stream of gas flowing through a de-polymerized zone. In this invention, the inert gas, hot carbon black, solid waste and heavy hydrocarbon off-specs oil are introduced into one end of the de-polymerized reactor and rapidly intermixed, physically in contact with each other and propel through the vessel radially to permit the requisite heat transfer to occur. The heat required to de-polymerize the organic matter is provided endothermic through the jacketed reactor by the Energeia heat catalyst system; that is contractually assigned and designed only for the utilization of heat transfer in this particular de-polymerization system. After initial heating up of the mixture, additional heat to remove the volatile chemical value in the rubber chip can be provided in part from the sensible heat in the carbon black and heavy fraction of the off-specs oil. Besides coming in contact with the waste solid rubber chip; the heat capacity and density of the hot carbon black and heavy fraction off-specs oil in the radially turbulent gaseous stream create an efficient zone of heat transfer. This is viable due to the unique contraption design of the propelling arms in clockwise and/or anti-clockwise within

the reactor. The system of this invention is based on a closed loop system; thereby the effluent from the de-polymerized zone of carbon black, volatized organic fuel and chemical values, water (as steam); inert gas and product gas can be substantially separated from the de-polymerized vapor by a unique clarification-centrifugation-purification for off-specs hydrocarbon oil, carbon black and steel wire. All effluent is reflux into the de-polymerized cycle as part of the end products.

Rubber chip (organic matter) is conveyed via a scraper conveyor (1) into a volumetric conveyor (2) thru a rotary airlock and into a cylindrical reactor (3) with propelling arms attached to the center shaft. The stationary reactor has a jacket that permit endothermic heating of the organic solid within the reactor. Upon vaporization of the mixture of organic solid at a temperature of 300-450 degree Celsius, the de-polymerized vapor exit from the two opening at the top surface of the reactor and travel along the pipework into the condenser-quencher (4). Quenching, in effect condensed at least a portion of the de-polymerized vapor into a heavy fraction off-specs liquid containing entrained particulate matter. The vaporized hydrocarbon, containing entrained particulate matters, travel along a network of pipes and can be further condensed in condenser (5) and condenser-cooler (6). The medium heavy fraction off specs oil is condensed at 80 degree C and further condensed-cooled at condenser (6) and is transfer to clarification tank (7), mixed and clarified with effluent water to be discharge via pipeline into a centrifuging-purifying system (8); where at a temperature of about 60 degree C, the medium heavy fraction liquid is converted into a light fraction off-specs hydrocarbon oil and stored at a light fraction off-specs tank (9). The light fraction off-specs oil is reflux via a piping system and into quencher-condenser (4) where the light fraction liquid is used in scrubbing at least a portion of de-polymerized hot vapor from reactor (3). The, at least a portion of heavy fraction hydrocarbon off-spec oil is discharge from the quencher-condenser (4) into a heavy fraction tank (10); where the heavy fraction liquid containing entrained particulate matter is refluxed into the reactor; and mixed with the carbon black, organic solids and hot gaseous inert and resident gases; to repeat the de-polymerized cycle. The

residue solid "carbon black" is propel toward the end of reactor (3) and exit via a vibrator chute; conveyed by a propelling paddle enclosed conveyor (11) via an airlock and drop into a conveyor (12) with propelling paddle; into a magnetic drum (13) where the carbon black is magnetically separated from remnant of steel wire; into uplifting conveyor (14) and finally into a carbon storage silo (15). The steel wire is separated by the magnetic drum and deposited via a belt conveyor into a transfer bin.

CLAIMS

1. A unique clarification-centrifugation-purification process for producing organic off spec crude and solid residue "carbon black" from rubber chips from discarded waste scrap tires, the process comprising the steps of:

(a) De-polymerization a solid organic waste, namely vulcanized rubber chips in a substantially N_2 inert, oxygen-free atmosphere to produce off-spec crudes vapor and a solid residue "carbon black" including fine particulate matter entrained in the de-polymerized vapor;

(b) Separating the de-polymerized off-spec crudes vapor containing entrained particulate matters from the solid residue;

(c) Condensing at least the first portion of the separated de-polymerized off-spec crudes vapor to produce de-polymerized off-spec crudes heavy fraction of American Petroleum Institute (API) gravity < 10°, heavy off-spec crude;

(d) Clarifying and centrifuging such de-polymerized off-spec crude to produce a medium heavy fraction of American Petroleum Institute (API) gravity < 29°, heavy off-spec crudes, substantially free of entrained particulate matter.

2. A process as recited in claim 1 wherein the de-polymerized off-spec crudes is quenched to produce an off-spec crudes heavy fraction containing entrained particulate matter; and said off-spec crudes medium heavy fraction condensed and cooled to produce the off-spec crudes medium heavy fraction substantially free of entrained particulate matter.

3. A process as recited in claim 2 wherein the off-spec crudes medium heavy fraction is mixed with hot water and centrifuge into an intermediate /Medium off-spec crude fraction of American Petroleum Institute (API) gravity < 39°, heavy off-spec crude.

4. A process as recited in claim 2 wherein the first off-spec crudes heavy fraction is mixed with the rubber chips in the reactor.

5. A process as recited in claim 4 wherein the first liquid portion of the de-polymerized vapor is recycled as a quench fluid to produce de-polymerized off-spec crudes vapor containing entrained particulate matter.

6. A process as recited in claim 1 comprising of forming a mixture of the off-spec crudes heavy fraction containing entrained particulate matter and a solid residue of entrained particulate matter.

7. A process as recited in claim 2 further comprising the steps of condensing at least a second portion of the de-polymerized off-spec crudes vapor to produce a medium off-spec crudes heavy fraction American Petroleum Institute (API) gravity < 29°.

8. A process as recited in claim 2 or 7 further comprising the steps of distilling the de-polymerized off spec crudes into a lower fraction and one higher boiling fraction: forming an off-spec crudes heavy fraction of American Petroleum Institute (API) gravity < 10°, and to produce an off-spec crudes medium heavy fraction of American Petroleum Institute (API) gravity < 29°, heavy off-spec crude.

9. A process as recited in claim 9 wherein said lower boiling fraction has a boiling point of up to about 300 degree Celsius and wherein said higher boiling fraction has a boiling point of up to about 400 degree Celsius.

10. A process as recited in claim 9 further recovering a fraction of de-polymerized off-spec crudes having a boiling point greater than 300 degree Celsius.

11. A process as recited in claim 9 further recovering a fraction of de-polymerized off-spec crudes having a boiling point greater than 400 degree Celsius.

a. De-polymerization a solid organic waste in a substantially N2 inert, oxygen free atmosphere to deliver a de-polymerization off-spec crudes vapor containing entrained, fine particulate matter and solid de-polymerized solid residue;
b. Separating the de-polymerized off-spec crudes vapor containing entrained particulate matter from the solid de-polymerized residue;
c. Quenching at least a first portion of the de-polymerized off-spec crudes vapor to produce de-polymerized off-spec crudes heavy fraction liquid containing entrained particulate matter;
d. Condensing at least a second portion of the de-polymerized off-spec crudes vapor to produce an off-spec crudes heavy-medium fraction liquid containing entrained particulate matter;
e. Cooling the de-polymerized off-spec crudes heavy medium fraction liquid containing entrained particulate matter;
f. Centrifuging-Purifying the de-polymerized off-spec crudes heavy medium fraction off-spec crudes containing entrained particulate matter;
g. Recovering the de-polymerized off-spec crudes heavy medium fraction liquid substantially free of entrained particulate matter;
h. Recovering the de-polymerized off-spec crudes containing a relatively greater amount of entrained particulate matter and separating said fraction into a first portion of heavy off-spec crudes with entrained particulate matter and a second off-spec crudes portion containing a medium heavy fraction containing a lesser amount of particulate matter than the first portion;
i. Recovering the off-spec crudes heavy fraction containing entrained particulate matter;
j. Mixing the off-spec crudes heavy factor with the rubber chip in the reactor to form a mixture containing rubber chips and entrained particulate matter;
k. Cooling the off-spec crudes heavy medium fraction via a cooler condenser;

1. Centrifuging and purifying the off-spec crudes medium heavy fraction into a light medium fraction that contain a substantially lower amount of particulate matters;

m. Refluxing the light fraction off-spec crudes into condenser No.1 for scrubbing particulate matters of the de-polymerized off-spec crudes vapor and mixing with the off-spec crudes heavy fraction with entrained particulate matter;

n. Recovering the off-spec crudes light fraction from the centrifuging and purifying step.

12. A process as recited in claim 1 (c) further comprising the steps of recycling the third off-spec crudes portion of the centrifuged off-spec crudes light fraction as a quench fluid to condense at least said first portion of the de-polymerized off-spec crudes vapor.

13. A process as recited in claim 7 further comprising the steps of forming a mixture of intermediate boiling fraction produced by distilling the de-polymerized off-spec crudes vapor fraction, the second portion of the off-spec crudes medium heavy fraction at 350 degree Celsius.

14. A process as recited in claim 13 wherein the fractions upon distilling the off-spec crudes comprising a low boiling fraction of 80 degree Celsius and a high boiling fraction having a boiling point greater than 350 degree Celsius.

15. An organic hydrocarbon liquid product from solid organic vulcanized rubber waste by the process of claim 1.

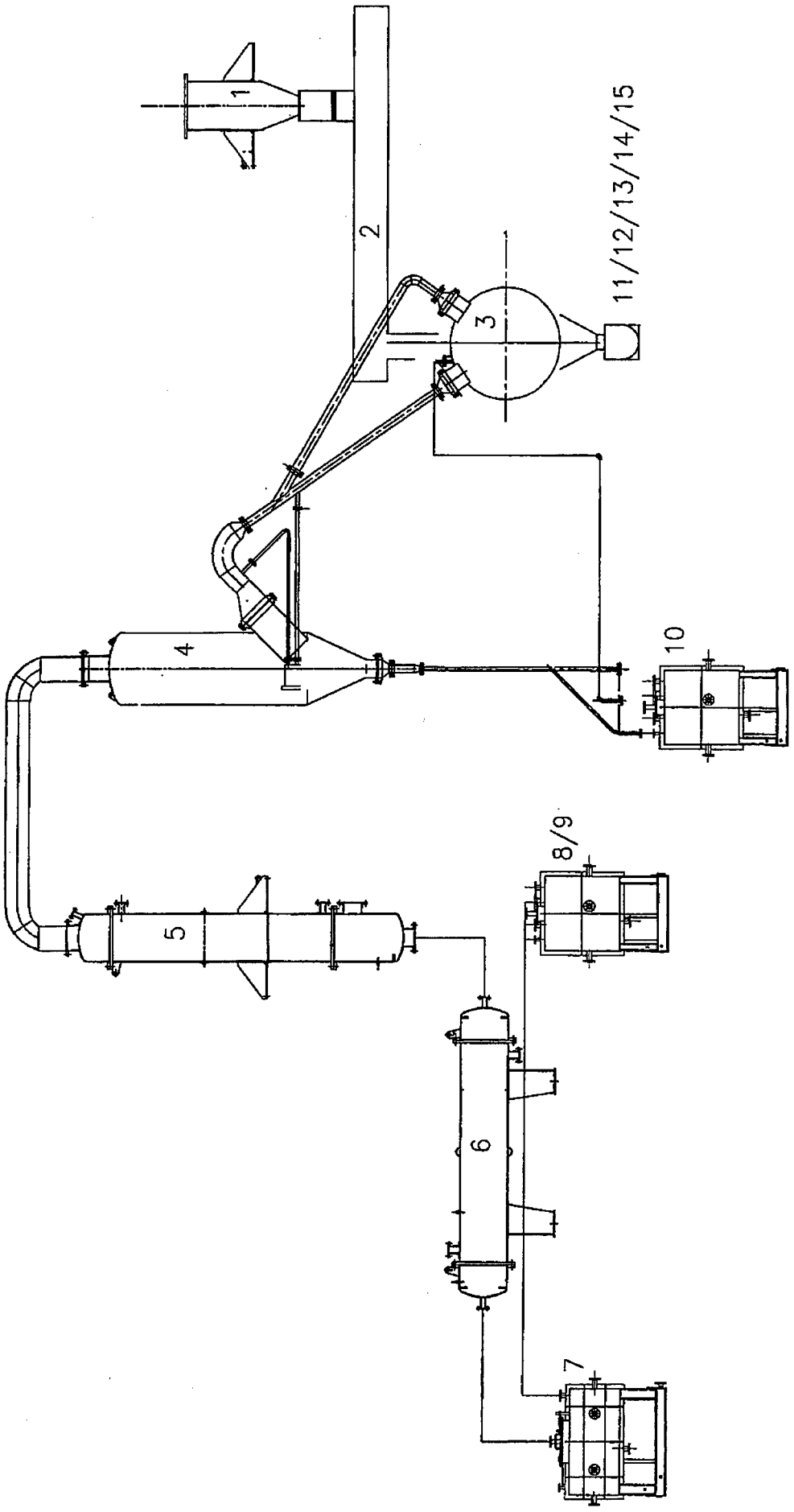

Figure 1

INTERNATIONAL SEARCH REPORT

International application No
PCT/MY2012/000280

A. CLASSIFICATION OF SUBJECT MATTER
INV. C09C1/48 C10G1/10 C10B53/07
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)
C09C C10G C10B

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 2007/102279 A1 (NOVAK JOHN F [US]) 10 May 2007 (2007-05-10) claims 1-9 paragraphs [0003] - [0005]	1-15
X	US 2008/179257 A1 (CLARKE STEPHEN [CA]) 31 July 2008 (2008-07-31) claims 1-24 paragraphs [0006] - [0018]	1-15
A	US 2010/249353 A1 (MACINTOSH ANDREW D E [CA] ET AL) 30 September 2010 (2010-09-30) claims 1-27	1-15
X	DE 24 00 284 A1 (AGENCY IND SCIENCE TECHN; NIPPON ZEON CO; JAPAN GASOLINE) 18 July 1974 (1974-07-18)	15
A	the whole document	1-14

☐ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

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"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
18 February 2013	25/02/2013

Name and mailing address of the ISA/	Authorized officer
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1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/MY2012/000280

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