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(54) PROCESS FOR REGENERATION OF WASTE OILS

(57) A process for the regeneration of waste oils is disclosed, comprising a fractioned distillation in a packed column (21) and hydrorefining and grinding of bitumen and any sediment from other steps, said grinding yielding a liquid fraction, collected as product, and a solid fraction, recycled to the first process steps. The oil to be regenerated is then fed to the process divided into two separate fractions, a better-quality one and a lower-quality one, containing more impurities, said better-quality fraction undergoing flash distillation, centrifugation and dehydration, to then be sent to the distillation packed column (21), while said worse-quality fraction undergoes homogenisation, grinding and decanting, the liquid phase which is obtained following these operations being mixed with the better-quality fraction and the solid phase being recycled to the homogenisation step.

A plant for the implementation of the process is also disclosed. Such a plant comprises a packed distillation column (21) and a reactor (27) for hydrorefining and has two feeds (1; 2) of oil to be regenerated and at least one grinding unit (5) of the solid sediments and at least one centrifugal separator (13).

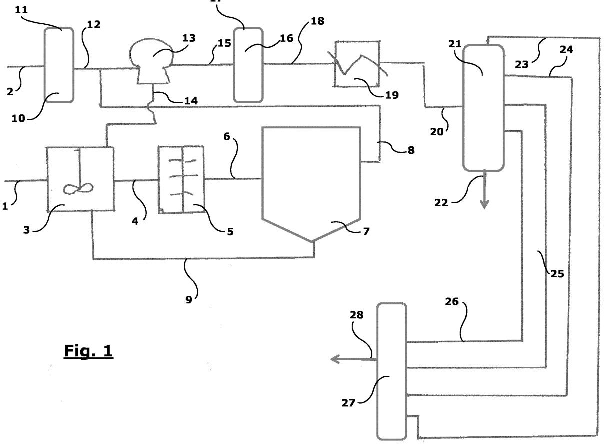

Fig. 1

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Description

[0001] The present invention relates to a regeneration process for waste oils, in particular mineral oils, aimed at achieving the enhancement of the same oils, with a lower power consumption compared to conventional processes.

[0002] Lubricating oils have been used for a very long time in almost all the fields of mechanics to allow engine parts or the like to slide on each other. A classic example is that of oil used in internal combustion engines to help the sliding of the pistons in the cylinders, without the mechanical parts getting stuck into one another.

[0003] Lubricating oils are often used in machinery involving very fast relative sliding movements of their parts. Because of this, in addition to other aspects, lubricating oils frequently undergo temperature changes, which are in some cases remarkable. For this reason, oils undergo chemical reactions of various kinds and their nature is altered. As a consequence, reactions such as cleavage, dehydration, dehydrogenation, condensation, etc. take place. An example of such a reaction is the dehydrogenation-dehydration of oils, which leads to the formation of carbon in the form of carbon black. In some cases, the formation of asphaltenes and bitumens also occurs. Furthermore, the oils can contact other substances, such as, for example, metallic and/or ceramic particles, thus remaining inside the oil itself. Moreover, it is not uncommon that spurious substances, such as various types of polymers, get mixed with oils, since their use and/or collection after use do not always take place in an appropriate manner and with precautions aimed at avoiding the introduction of foreign substances and obtaining a quality waste.

[0004] The above-mentioned and other transformations determine the deterioration of the physical and chemical features of the oil, making it eventually harmful for the machines inside which it is used, because the foreign substances inside the oil significantly increase friction. Therefore, it is appropriate to replace the oil with fresh oil after a certain period of use, so as to avoid performance reduction ore even breakage of mechanisms.

[0005] Waste oil normally contains a number of toxic substances, for which reason it cannot be simply discharged into the environment, but must be treated to reduce its environmental impact. The regeneration of waste oils has thus emerged as a solution allowing to reduce waste and the resulting environmental impact.

[0006] The first plants for re-refining waste oils date back to the 60s of the twentieth century, when environmental awareness began to develop and spread. Given the high costs to obtain freshly refined oils and because of the increase in consumption of these oils, the regeneration of waste oils has been gaining important market shares over time.

[0007] The establishment of mandatory consortia responsible for the collection of waste oils has also made easily available the material to be treated - the actual raw material of these processes -, now widely accessible at a good price, although its quality is sometimes quite poor; in particular, there are types of quality waste which can be quite different from each other. This type of market has significantly expanded over the years. About a third of waste oil is properly collected by the consortia and sent to regeneration or to other treatments to make it harmless, the rest being improperly dispersed in an uncontrolled manner into the environment and being a source of pollution or, in any case, lost in its service cycle, with a remarkable economic damage as well.

[0008] The typical yield of a regeneration process, starting from 100 kg of waste oil to be sent to the process, is now always around 60 kg of regenerated oil (base for lubricants), 20-25 kg of fuel and 20-25 kg of bitumen.

[0009] Historically, the first processes were those with sulphuric acid or with liquid propane. In other words, oils are treated by adding sulphuric acid or propane, so as to eliminate a large part of impurities contained in waste oils. However, the process involving the use of acid has been virtually abandoned, due to the considerable pollution problems it entails.

[0010] The process with sulphuric acid causes the formation of acid sludge, which retains non-negligible amount of oil and also contains polymeric compounds and heavy metals. This sludge is difficult to dispose of; its disposal generally occurs in landfills, preferably after neutralisation, which, however, increases the volume of the sludge itself. The solution of sludge combustion has been tested, but turned out to be impractical.

[0011] Other processes, starting from the already mentioned one that makes use of propane, were thus developed.

[0012] The step of acid treatment is completely replaced by clarification with liquid propane. The chosen hydrocarbon is propane for being easily liquefiable and having a low density once liquefied. It therefore acts as fluidifying agent on oils to which it is added, so as to allow the separation of a high-density phase - containing high molecular weight polymers and heavy metals - from a second fraction, constituted by clarified and dehydrated oils. The propane is then removed and recycled by mixing with the in-fed oils.

[0013] A hot filtration is then performed, allowing the recovery of a gaseous fraction. Discoloration and deodorisation of the content are the final steps.

[0014] However, this process allowing to achieve higher yields still has some disadvantages. First of all, handling propane can be dangerous for plant workers. Moreover, a part of the propane is trapped in the asphalt; this presence excludes the use of the obtained bitumen for the construction of roads, with considerable market limitation and consequent economic damage. As a result, the bitumen fraction in this process is simply a waste to dispose of and cannot be enhanced. Finally, the process itself is much more expensive than the process with sulphuric acid.

[0015] In more recent years, a new type of process has been introduced, according to which waste oils are sent to the axial rotor of a distillation column and are sprayed by said rotor on the inner walls of the column, kept at a high temperature by a diathermic fluid flowing through some ducts. In contact with the heated walls, the vaporisable fraction evaporates, while the heavier fraction remains in the liquid phase. Fractions are then collected and further fractioned in a subsequent fractioning column, which is divided in further cuts inside a subsequent fractioned distillation column. The yields of this process are important, but a pre-treatment is required to purify the waste oils.

[0016] EP 0 618 959, of the same Applicant, discloses a process for re-refining waste oils, in which said oils are contacted with a basic reagent and heated to remove contained water, polymers and heavy metals are separated and fractioned distillation is performed in a packed column, in order to obtain one or more base fractions for lubricant, followed by discoloration. The basic reagent is a strong base, water is removed together with a more volatile fraction in a preliminary step of flash distillation, while polymers and heavy metals are removed mostly by decanting. However, the packed distillation column tends to become clogged by solid residues, still contained in the oil to be fed inside the same. Furthermore, the flash distillation column entails high operating costs.

[0017] Recently, the same Applicant filed the Italian patent application no. MI2015A 000626 for a process which, starting from that of EP 0 618 959, provides for a centrifugation step of the oil to be regenerated between the step of flash distillation and the step of packed column distillation. The pressure inside the packed distillation column is adjusted through a liquid-ring pump, obtaining a cleaner product and a reduced number of interventions to regenerate the distillation column.

[0018] WO2004/033 608 discloses a process and a device for the treatment of waste oils, which includes a preliminary separation by decanting of at least one fraction of the water and one fraction of solid particles (sediments), a pre-heating of the oily phase from the decanting step and centrifugal separation of the oil, preheated to a temperature below the boiling point of water, followed by the separation of water and other pollutants. The product obtained, however, is not reused to produce new lubricant bases, but is fed to a combustion step, along with other hydrocarbons, therefore with no enhancement of the same.

[0019] WO96/00 273 discloses a process of reclamation of waste oils, with a view to their reuse. According to this document, waste oil undergoes a centrifugation as it is to remove solid matters; the output oil is then contacted with diammonium phosphate and/or oxalic acid at a temperature between 60 and 85-90°C and is then subjected to a new centrifugation to separate oil and water. There are no distillation steps.

[0020] In all the processes just examined, next to the lubricant base - which is the most refined product - important quantities of bitumen and asphalt are obtained and normally sold for basic uses, such as the construction of paved roads.

[0021] In order to enhance the bitumen fraction as well, the Applicant has filed, simultaneously with the patent priority of which is here claimed, an Italian patent application referring to a process for the regeneration of waste oils, comprising a flash distillation step of oil to be regenerated, a decanting step of the heavy fraction, a distillation step in a packed distillation column, allowing to produce an oil fraction to be sent to hydrorefining and a bitumen fraction. In this process, the bitumen fraction output as a tail from the distillation step is ground, the liquid fraction that is obtained after this grinding being collected as bitumen and the remaining solid fraction being recycled to the decanting step.

[0022] In all of the above-mentioned processes, there is a relatively high power consumption and it would be desirable to reduce it, even by the slightest degree.

[0023] FR 2 787 118 discloses the recovery of high value-added additives, while enhancing the distillation tails, resulting in a high viscosity index oil by low-temperature fractioned distillation.

[0024] DE 2 605 484 discloses a process for making waste mineral oil reusable, which involves heating the oil to a temperature of 204-427°C, at a pressure between 35,2 and 246 kg/cm^2 and thereafter separating the foam that forms.

[0025] DE 69 524 533 relates to a process for the purification of a waste mineral oil, comprising the steps of dehydration, vacuum distillation, solvent extraction and hydrorefining. Dehydrated oil is directly distilled under vacuum; the distillation tail is subjected to extraction with solvents; the output from these treatments is subjected to hydrorefining.

[0026] JP 1988 0 291 191 describes a treatment process of oily residues in a steel mill, involving grinding of steel mill residues separated from oil and their combustion to produce ferric oxide.

[0027] The object of the present invention is to propose a process allowing to obtain lubricant bases and good quality bitumen, possibly reducing the required power consumption, thus overcoming the drawbacks of the prior art. This task is achieved, according to the present invention, by a process for the regeneration of waste oils, which provides a fractioned distillation in a packed column and hydrorefining of the oil and the grinding of sediments, characterised in that the oil to be regenerated is fed divided into two separate fractions, a better-quality one and a lower-quality one containing more impurities, said better-quality fraction undergoing flash distillation, centrifugation and dehydration, to be subsequently sent to the distillation packed column, while said worse-quality fraction undergoes homogenisation, grinding and decanting, the liquid phase which is obtained following these processes being mixed with the better-quality fraction and the solid phase being recycled to the homogenisation step. The subclaims disclose further preferred embodiments.

[0028] The present invention is now described in greater detail, making reference to the accompanying drawings, wherein:

fig. 1 is a diagram representing a first embodiment of the present invention;
fig. 2 is a diagram representing an alternative embodiment of the present invention; and
fig. 3 is a block diagram representing a part of a further alternative embodiment of the present invention.

[0029] A first embodiment of the present invention is represented in fig. 1. According to the present invention, the oil to be regenerated is divided, at the time of acquisition, into two fractions, a better-quality one and a worse-quality one. Normally, the worse-quality fraction contains substantial quantities of foreign matter, such as, for example, polymers and similar pollutants, while the better-quality fraction exhibits a low or even negligible content of solid matter, such solid substances being mostly carbon residues, related to oil impairing in its use. In particular, for the purposes of the present invention, the better-quality oil fraction to be regenerated has a total sediment of a maximum of 3% by volume, a minimum viscosity at 50°C of 1.8°E, a maximum sulphur content of 1.5% by mass, while the worse-quality oil fraction to be regenerated has a total sediment in an amount greater than 3% by volume, a minimum viscosity at 50°C of 1°E and a maximum sulphur content equal to 3% by mass.

[0030] The worse-quality fraction is fed to the plant at 1, while the better-quality fraction is fed at 2. The fraction 1 then enters a mixer 3. The mixer 3 mixes and homogenises the incoming oil, so as to disperse the impurities within it in a uniform manner, thus reducing to a minimum the possible phase separations. In view of this, it is possible to arrange an emulsion treatment. The mixed and homogenised fraction output from the mixer 3 is sent to a grinding device 5 through pipeline 4. The grinding device 5 can be of any known type. Instruments such as ball mills and wedge mills are preferred. Preferably, said grinding device 5 is a ball mill, easy to use and allowing to achieve excellent results. The device 5 breaks and opens solid particles contained in the oil to be regenerated, thus making available more oil, contained within the particles themselves. A pipeline 6 sends the ground fraction to a decanter 7. The floating part in the decanter 7, essentially containing a liquid, exits through a pipeline 8, while the part on the bottom of the same decanter 7, containing mainly solid sediments, therefore pollutants, exits through a pipeline 9 which brings it back to the mixer 3.

[0031] Regarding the better-quality fraction 2, it is, first, fed to a flash distillation column 10. The column 10 is operated at a pressure of about 250 torr and a temperature of about 130-140°C, slightly higher than the water boiling temperature, to remove as much water as possible from the oil to be regenerated. The aqueous fraction leaves the column 10 through the outlet 11. Instead, the fraction containing the oil to be regenerated comes out of the pipeline 12, in which the liquid from the pipeline 8 flows, and is fed to a centrifugal separator 13. The centrifugal separator 13 outputs two flows: a flow 14, containing mostly solids sediment, which is sent to the mixer 3, and a second flow 15, containing the fluid to be regenerated, that is sent to a dehydration unit 16. Water exits the unit 16 through a flow 17, while the oil to be regenerated exits from a pipeline 18. The drawing shows only one centrifugal separator 13 and only one dehydrating unit 16, but it is obvious that there may be more of them, either in series or in parallel, without departing from the scope of the present invention.

[0032] The oil to be regenerated inside the pipeline 18, before being subjected to fractioned distillation, can optionally be subjected to adjustment of its viscosity, according to processes known per se to the person skilled in the art, thus improving its commercial impact. The pipeline 18 is fed to a heat exchanger 19, preferably an oven, bringing the oil temperature to a value higher than 300°C, preferably to a value between 350 and 400°C. Before entering the oven 19, the oil to be regenerated can be sent to a further centrifugal separator, not shown in the figures. The sediments output from this additional centrifugation step are preferably sent to the mixer 3. A pipeline 20 carries the heated oil to a packed distillation column 21, where fractioned distillation of the oil is performed. The pressure inside the column 21 is adjusted, preferably, to a value ranging from 8 to 15 torr. In a preferred variant, in said packed distillation column 21, pressure is adjusted by a liquid-ring pump, allowing to reduce the clogging of the packing. The tail of the column 21 produces a fraction 22, containing mostly bitumen, which can be sold or used as such.

[0033] Column 21 also outputs head fractions 23 and intermediate fractions 24, 25 and 26. The fractions 23, 24, 25 and 26 are then sent to a reactor 27, where a hydrorefining step is performed, yielding the desired lubricant bases, output at 28. In the processes according to the prior art, the reactor 27 is normally operated at a pressure above 80 bar. The fact of having separated the best and worst fractions at the beginning and having ground the sediment output from the decanter 7 and from the centrifugal separator 13 ensures that, with the process according to the present invention, one can obtain similar or even better results managing the reactor 27 at a pressure of about 60 bars, which implies a considerable power saving. In case the version of the column 21 comprising a liquid-ring pump is used, it is moreover possible to obtain a remarkable extension of the life of the catalyst contained in the reactor 27, the duration of which goes from an average of about 4 months to an average of about 8 months for its deactivation. There is also a reduction of required maintenance for the column 21. This provides considerable savings, since these catalysts are mostly based on precious metals.

[0034] According to a variant, the fraction 23, either subjected to hydrorefining or as it is, can be used within the system to operate a cogeneration engine that provides electric power and steam to the entire system.

[0035] The water from the plant, for example in 11 and 17, can be subjected to an oxidation treatment of its pollutants with wet air (technique known as wet air oxidation), thus allowing to recover steam, to be used in the plant.

[0036] An alternative embodiment of the present invention is represented in fig. 2. Components identical to those of the previous embodiment will carry the same reference numerals. The operating parts identical to those of the embodiment of Fig. 1 will not be described again to avoid encumbering the reading and, for them, it is possible to refer to what previously reported.

[0037] The only difference from the embodiment of Fig. 1 is that the bitumen, leaving the packed distillation column 21 through the pipeline 22, instead of being used or sold as such, is subjected to an enhancement process, which also has the effect to further push the yield in the lubricating bases produced. The pipeline 22 carries the bitumen to a mixer 29, which homogenises the bitumen. The bitumen comes out through a pipeline 30 and is fed by it to a grinding device 31, completely analogous to the mixing device 5, already seen previously. The liquid fraction, consisting of a more valuable bitumen which can be used for more refined uses than normal, as for the production of inks, comes out in 32, while the solid fraction is re-fed to the mixer 3, along with the other sediment and to the fraction 1, containing the oil of worse quality to be regenerated.

[0038] Another alternative embodiment is illustrated in Fig. 3. Even in this case, parts identical to those of the preceding embodiments have identical reference numerals. Even in this case, the common part of the description is omitted to avoid burdening the discussion.

[0039] Fractions 23, 24, 25 and 26, output from the packed distillation column 21, are fed to a tank 34 which acts as a buffer, to allow the subsequent operations to be carried out discontinuously, load-wise or batch-wise.

[0040] A pipeline 35 feeding a microfiltration unit 36 exits from the tank 34. Two flows are output by this unit. A pipeline 37 sends the oil fraction to the hydrorefining reactor 27, from which the produced lubricant bases will exit. A pipeline 38 removes the solid sediments from the unit. Optionally the pipeline 38, instead of the waste, can bring sediments to the mixer 3, increasing the quality of bitumen produced and its yield. Therefore, between the step of fractional distillation in a packed column and the step of hydrorefining, a microfiltration step of the oil in regeneration is provided.

[0041] The present invention allows to achieve important power savings, obtaining high yields and extremely clean products of high value. In particular, compared to the prior art processes, not all the oil to be regenerated is subjected to flash distillation, but only the better quality fraction. In this way, the column 10 can be of relatively small size and requires less power for its operation compared to the power that would be required to submit all the oil to be regenerated to flash distillation. Secondly, as seen previously, the pressure within the hydrorefining reactor is maintained much lower for the same performance, resulting in a lower power consumption for the maintenance of pressure.

[0042] The present invention also relates to a plant for the implementation of the process, this plant comprising a packed distillation column 21 and a reactor 27 for hydrorefining, characterised in that it has two feeds 1 and 2 of oil to be regenerated, a worse-quality fraction being fed to one (1) of said feeds 1, 2 and a better-quality fraction being fed to the other (2) of said feeds 1, 2 and in that it further comprises at least a grinding device 5 for grinding solid sediments through which the worse-quality fraction passes after the feed 1, and after passing through a mixer 3 and at least one centrifugal separator 13, in which a fraction 12 is fed, consisting of the oil fraction to be regenerated, coming from the better-quality feed 2 output by a flash distillation column 10, which is mixed with the part floating in a decanter 7 which received the fraction ground in the device 5.

[0043] The present invention has so far been disclosed with reference to its preferred embodiments, but is not limited to them, many variants being available to the person skilled in the art, without departing from its scope of protection, which is defined by the appended claims.

LIST OF REFERENCE NUMERALS

[0044]

1	Worse-quality fraction feed
2	Better-quality fraction feed
3	Mixer
4	Pipeline
5	Grinding device
6	Pipeline
7	Decanter
8	Pipeline
9	Pipeline
10	Flash distillation column
11	Output (of 10)
12	Pipeline
13	Centrifugal separator
14	Flow with solid sediment (from 13)
15	Liquid flow (from 13)
16	Dehydration unit
17	Water flow (from 16)
18	Pipeline
19	Heat exchanger
20	Pipeline
21	Packed distillation column
22	Bitumen fraction (from 21)
23	Distilled fraction (from 21)
24	Distilled fraction (from 21)
25	Distilled fraction (from 21)
26	Distilled fraction (from 21)
27	Hydrorefining reactor

28 Output (of 27)
29 Mixer
30 Pipeline
31 Grinding device
32 Bitumen
33 -
34 Tank
35 Pipeline
36 Microfiltration unit
37 Pipeline
38 Pipeline

Claims

1. Process for the regeneration of waste oils, which provides a fractioned distillation in a packed column (21) and hydrorefining of the oil and the grinding of sediments, **characterised in that** the oil to be regenerated is fed divided into two separate fractions, a better-quality one and a lower-quality one, containing more impurities, said better-quality fraction undergoing flash distillation, centrifugation and dehydration, to be subsequently sent to the distillation packed column (21), while said worse-quality fraction undergoes homogenisation, grinding and decanting, the liquid phase which is obtained following these processes being mixed with the better-quality fraction and the solid phase being recycled to the homogenisation step.

2. Process as in claim 1), **characterised in that** the better-quality oil fraction to be regenerated has a total sediment of a maximum of 3% by volume, a minimum viscosity at 50°C of 1.8°E, a maximum sulphur content of 1.5% by mass, while the worse-quality oil fraction to be regenerated has a total sediment in an amount greater than 3% by volume, a minimum viscosity at 50°C of 1°E and a maximum sulphur content equal to 3% by mass.

3. Process as in claim 1) or 2), **characterised in that** it also provides for a treatment of the emulsions during said homogenisation step.

4. Process as in any one of the preceding claims, **characterised in that** the part on the bottom in said decanting step, containing mainly solid particles, exits from a decanter (7) through a pipeline (9) which brings it back to the mixer (3).

5. Process as in any one of the preceding claims, **characterised in that** one flow (14) output from the centrifugation step and containing mostly solids sediments, is sent to the mixer (3), and a second flow (15) coming from the same centrifugation step is sent to a dehydration unit (16).

6. Process as in any one of the preceding claims, **characterised in that**, before undergoing fractioned distillation, the oil to be regenerated undergoes a viscosity adjustment step.

7. Process as in any one of the preceding claims, **characterised in that** the overhead fraction (23) output from the packed distillation column (21) is used within the plant to operate a cogeneration engine that provides electric power and steam to the entire plant.

8. Process as in any one of the preceding claims, **characterised in that** the water leaving the plant undergoes oxidation treatment of its pollutants with wet air (wet air oxidation technique), thus allowing to recover steam to be used in the plant.

9. Process as in any one of the preceding claims, **characterised in that** the bitumen output as the tail from the packed distillation column (21) is sent to a mixer (29), which homogenises the bitumen, to be then fed to a grinding device (31), the solid fraction being refed to the mixing step (3) of the worse quality fraction fed to the process.

10. Process as in any one of the preceding claims, **characterised in that**, between the step of fractioned distillation in a packed column and the step of hydrorefining, a microfiltration step of the oil in regeneration is provided.

11. Process as in claim 10), **characterised in that** the sediments output from the microfiltration step are fed to the mixing step (3) of the worse-quality fraction fed to the process.

12. Plant for the implementation of the process, according to any of the preceding claims, this plant comprising a packed distillation column (21) and a reactor (27) for hydrorefining, **characterised in that** it has two feeds (1; 2) of oil to be regenerated, a worse-quality fraction being fed to one (1) of said feeds (1; 2) and a better-quality fraction being fed to the other (2) of said feeds (1; 2) and **in that** it furthermore comprises at least a grinding device (5) of solid sediments through which the worse quality fraction passes after the feed (1), and after passing through a mixer (3) and at least one centrifugal separator (13), in which a fraction (12) is fed, consisting of the fraction containing the oil to be regenerated, coming from the better feed (2) output by a flash distillation column (10), which is mixed with the part floating in a decanter (7) which received the fraction ground in the device (5).

13. Plant as in claim 12), **characterised in that** the better-quality oil fraction to be regenerated has a total sediment of a maximum of 3% by volume, a minimum viscosity at 50°C of 1.8°E, a maximum sulphur content of 1.5% by mass, while the worse-quality oil fraction to be regenerated has a total sediment in an amount greater than 3% by volume, a minimum viscosity at 50°C of 1°E and a maximum sulphur content equal to 3% by mass.

14. Plant as in claim 12) or 13), **characterised in that** said grinding device (5) is a ball mill.

15. Plant as in any one of claims 12) to 14), **characterised in that**, inside said packed distillation column (21), pressure is adjusted by a liquid-ring pump.

16. Plant as in any one of claims 12) to 15), **characterised in that** it comprises a second grinding device (31) for the bitumen output by the packed distillation column (21).

17. Plant as in any one of claims 12) to 16), **characterised in that** it also comprises a microfiltration unit (36).

18. Plant as in any one of claims 12) to 17), **characterised in that** between the dehydration unit (16) and the packed distillation column (21), a further centrifugal separator is provided.

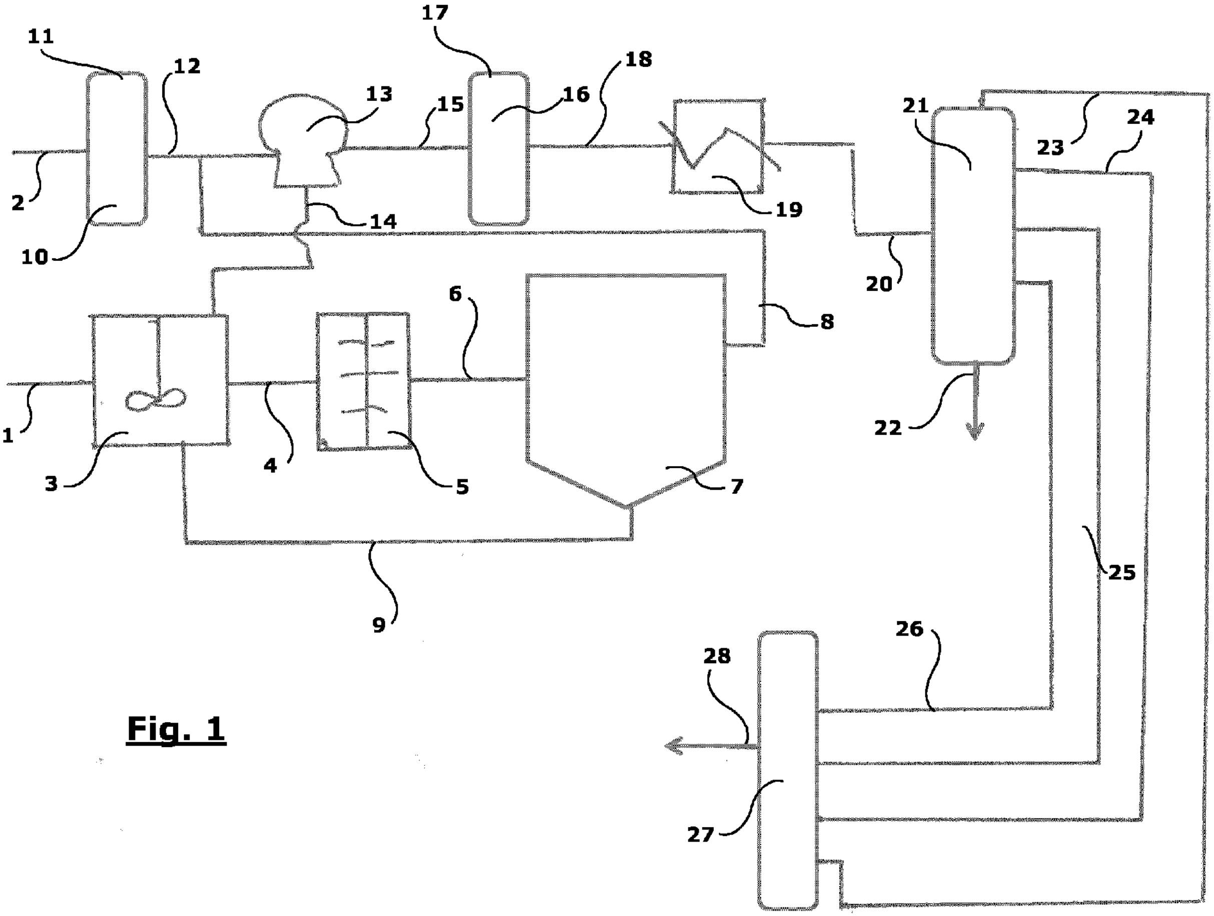

Fig. 1

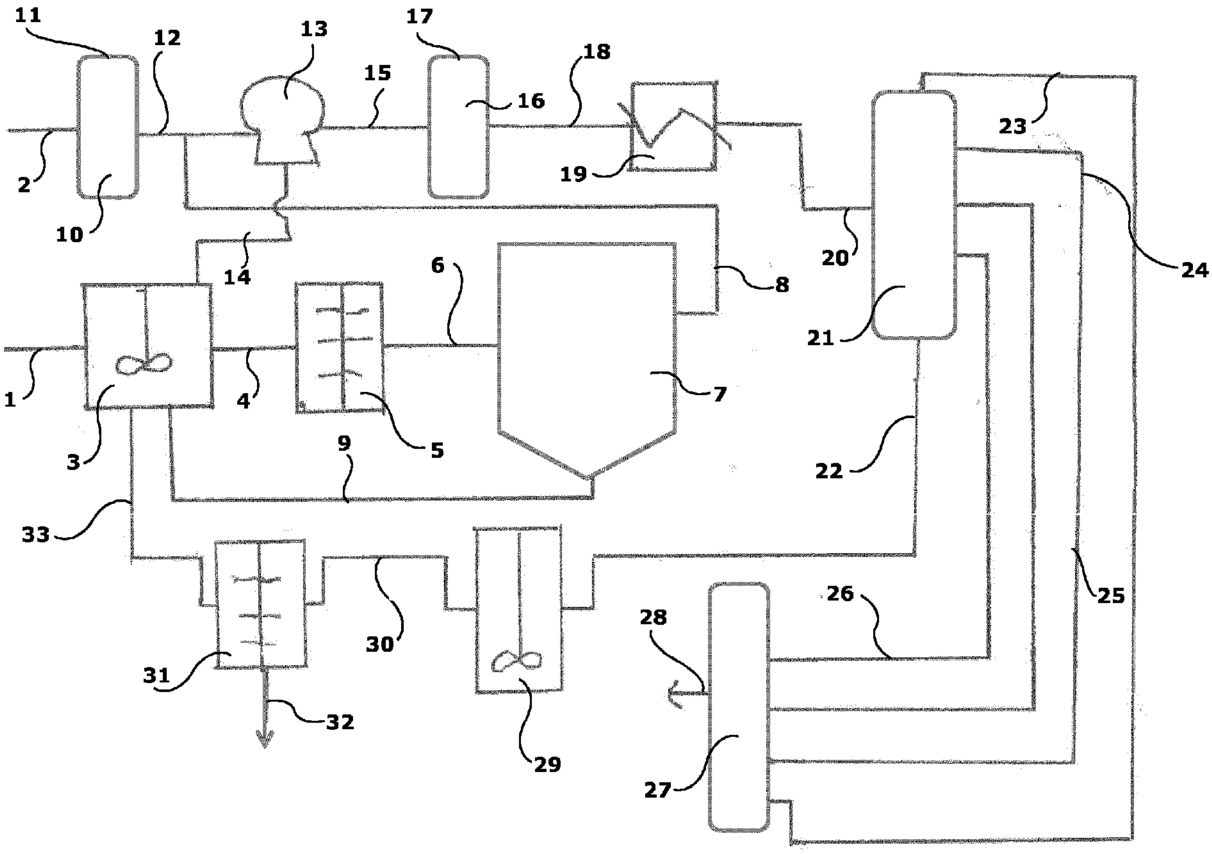
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Fig. 2

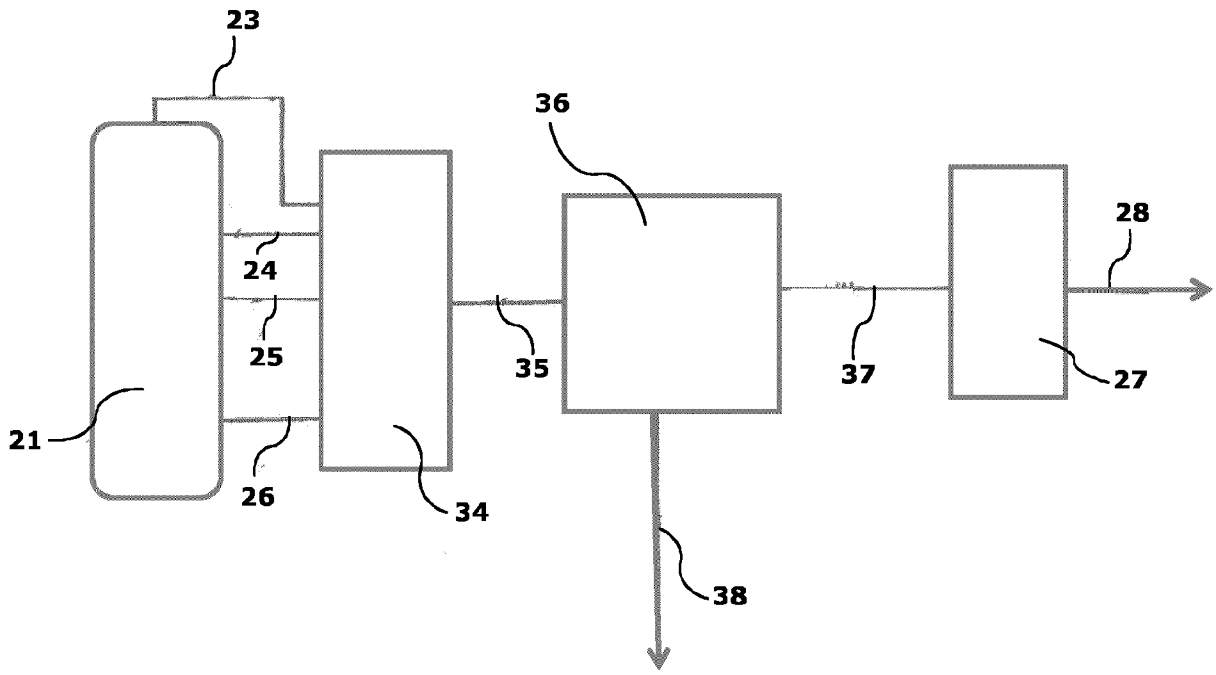

Fig. 3

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European Patent Office
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EUROPEAN SEARCH REPORT

Application Number
EP 16 16 4483

DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
A	FR 2 787 118 A1 (DEUTSCH RICHARD [FR]) 16 June 2000 (2000-06-16) * page 1 - page 5; claims 1,3,5,6,9,10 *	1-18	INV. C10M175/00
A	DE 26 05 484 A1 (WITCO CHEMICAL CORP) 11 November 1976 (1976-11-11) * page 6 - page 7; claims 1,3; examples B,C,D *	1-18	
A	DE 695 24 533 T2 (INST FRANCAIS DU PETROLE [FR]) 29 May 2002 (2002-05-29) * page 4 - page 12; figure 1 *	1-18	
A	DATABASE WPI Week 199028 Thomson Scientific, London, GB; AN 1990-214349 XP002751241, & JP H02 145437 A (MITSUBISHI HEAVY IND CO LTD) 4 June 1990 (1990-06-04) * abstract *	12-18	
			TECHNICAL FIELDS SEARCHED (IPC)
			C10M

The present search report has been drawn up for all claims

1

Place of search	Date of completion of the search	Examiner
Munich	9 August 2016	Pöllmann, Klaus

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
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E : earlier patent document, but published on, or after the filing date
D : document cited in the application
L : document cited for other reasons
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EPO FORM 1503 03.82 (P04C01)

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

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