

PATENT SPECIFICATION

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(54) SEPARATOR FOR TWO IMMISCIBLE LIQUIDS

(71) We, EXXON RESEARCH AND ENGINEERING COMPANY, a Corporation duly organised and existing under the laws of the State of Delaware, United States of America, of Linden, New Jersey, United States of America, do hereby declare the invention for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:-

The present invention concerns a separator operating on gravity and suction and using filters.

15 This separator is designed for the separation of mixtures of two non-miscible liquids having different densities, in particular, mixtures of water and hydrocarbons.

Purification equipment is already known 20 for water polluted by hydrocarbons, using separators working by gravity to obtain the separation of constituents in the different densities. The degree of purity of the outgoing water depends greatly on the difference 25 of density between the constituents, on the emulsion of the hydrocarbon droplets, on the quality of sediment attached to the droplets and increasing the apparent density, and eventually on the detergent content.

30 The degree of purity of the water obtained with the known separator, which is expressed in parts per million (p.p.m.) is, in general, not lower than 50 ppm or even 100 ppm when the hydrocarbon is very emulsified. 35

The present invention aims therefore at remedying this inconvenience, inherent to known separation processes, by creating a separator which, despite its reduced volume, 40 allows purity degrees of a few ppm only to be reached.

The present invention comprises a separator suitable for the separation of a first liquid and a second liquid having a 45 higher specific gravity than that of the first liquid from a liquid mixture containing both liquids, the liquids being mutually immiscible. The separator comprises an inlet for introducing the mixture into a first separation 50 zone so constructed and arranged as to

promote the separation by gravity of a substantial part of the first liquid from the mixture, a first liquid outlet for the discharge of separated first liquid from the separator, and a second liquid outlet for the discharge 55 of separated second liquid from the separator. There are means for conducting first liquid separated in the first separation zone to the first liquid outlet, means for conducting the remaining liquid mixture 60 containing a minor proportion of the first liquid and a major proportion of the second liquid towards the second liquid outlet along a flow path including a part extending downward through a second separation 65 zone comprising a liquid-permeable bed of filter material which is pulverulent or granular and which is active for retaining and coalescing droplets of the first liquid. There are means for causing a pressure reduction 70 across the second separation zone, so that liquid is drawn through the second separation zone under reduced pressure and means for conducting the resulting liquid 75 from the bottom of the liquid-permeable bed to the second liquid outlet.

In accordance with a preferred embodiment of the invention the separator comprises a tank. A dome shaped upper part of this tank is fitted with a vent and accommodates the feed-pipe for the mixture to be separated and the rod of a float. A frusto conical shaped lower part of the tank is fitted with a sediment outlet pipe and outlet pipe for the purified second liquid. The tank 85 possesses internal partitions which form a pre-separator and as the first separation zone a gravity separator. The partially treated second liquid flows out reaching the free surface at atmospheric pressure under 90 the dome, and the separated first liquid drains under gravity, since the tank also contains a pre-filter and a filter with a filtration media having coalescent power, operating under vacuum created by a pump fitted in 95 the outlet pipe.

Various other features of the invention can be seen in the following detailed description.

One preferred form of the separator is 100

represented on the accompanying drawings in which:

Fig. 1 is a vertical section of a sketch of a separator according to the invention

5 Fig. 2 is a cross-section following the line II-II on Fig. 1

Fig. 3 is a cross-section following the line III-III on Fig. 1.

The separator illustrated on Fig. 1 consists of a cylindrical envelope 1 with vertical axis and forming a tank, the upper part 2 of which is dome shaped, and is fitted with a vent 3, the mixture feed pipe 7 and the rod 8 of a float, controlling a suction pump 9 situated at the separator outlet. A pipe 5 fitted with a valve 6, for the periodical draining of sediments, is connected to the cone shaped lower part 4 of the envelope. The latter contains two cylindrical envelopes 10, 11 concentric to the vertical axis of the envelope 1 and at a certain distance from each other. These envelopes 10, 11 are open at their upper parts and encircle a central tube 12 which, at its lower end from a certain level is itself encircled, leaving an annular space, by a pipe 18 of bigger diameter. The latter is connected via a valve 40 to the suction pump 9.

The cylindrical envelopes 1 and 10 form an annular volume 13 serving as a gravity pre-separator, which has a widened portion at the top forming the hydrocarbon collecting compartment 15, as is clearly shown in Fig. 2. This collecting compartment 15 is bounded by an auxiliary vertical partition 14 represented by a dotted line of Figure 1 and a full line in Fig. 2. This partition 14 extends around approximately half the circumference of the envelope 1. A horizontal partition 16 constitutes the bottom of the collecting compartment 15. The upper edge of the auxiliary partition 14 is used as an overflowing edge for the hydrocarbon layer separated in the pre-separator 13 and flowing into compartment 15. The hydrocarbons so collected are discharged via pipe 17.

The cylindrical envelopes 10, which is prolonged at its lower end by a truncated cone 19, the cylindrical envelope 11 as well as the tubular conduit 18 define several annular volumes by successive partitions 20, 21 and 22 and successive conical partitions 23 and 24.

The inlet collector 41 situated at the lower end and constituted by the annular volume defined by the truncated cones 19 and 24 and partitioned by the radial baffles or fins 43 guides the water streams and coalesces of the oil (see Fig. 3).

60 Above the annular collector 41, the volume 42, forming a gravity separator, is defined by truncated cone 24, the tubular conduit 18, the cylindrical envelope 11 and the lower face of the truncated cone 23. This gravity separator 42 is extended upwards by

the roughly rectangular vertical chimney 25 the upper edge of which constitutes the overflowing weir 26 for the recovery of the hydrocarbon flowing to the collecting compartment 15.

The volume of the gravity separator 42 also communicates with an annular water discharge chimney 27 formed by the cylindrical envelope 11 and the external wall of the central tube 12. The latter is terminated at its upper end by a truncated cone the open edge of which 28, constitutes an overflow weir for water and a reference level for the liquids present in the pre-separator 13 and in the separator 42.

The pre-filter 29 is placed in the upper part of the separator and inside a compartment formed by the horizontal perforated partitions 20 and 21. This filter consists of a pulverulent media (sand, gravel, plastic granules or equivalent) forming a filtering bed which by its granulometry opposes the passage of particles making up the filter during the counter flow de-clogging and washing operations of this filter described below.

The middle section contains the actual filter 30 which is placed within the space formed by the horizontal partitions 21 and 22. Partitions 22 is fitted with a great number of mushroom shaped sieves drilled with holes of a diameter smaller than the gauge of the holes of the filter bed partitions, the role of which is essentially to distribute and diffuse the injected mixture of water and compressed air during the counter flow washing of filter 30. In the case of hydrocarbons separation, the filter bed is made of oleophilic material with fine granulometry, occupying approximately two-thirds of the compartment height. The oleophilic materials which are most preferred for the separation of hydrocarbon oils from water are complexes formed by an amine bound to a substrate through a binding reagent which is preferably an acid chloride. Useful substrates include untreated clays such as kaolinite, bentonite or montmorillonite or crushed bricks or crushed glass or glass beads. Suitable amines include aliphatic amines containing 6 to 20 carbon atoms per molecule or araliphatic amines or aromatic amines.

The material most preferred as the powdered filter material for the separation of hydrocarbons from water is that sold by Creusot-Loire under the Trade Name "Materiel Oleophile Creusot-Loire"

Filter 30 is situated above a volume formed by horizontal partition 22 and the truncated cone 23, which constitutes the water collector 32. The truncated cone 23 is terminated at its lower end by the discharge orifice 33, opening into the annular water discharge conduit 34 which is defined by the conduits 12 and 18. This conduit 34 is con-

5 nected via valve 40 the self-priming suction pump 9 discharging the sucked water into conduit 35. Between the separator and the pump three conduits 36, 38 and 39 are connected to conduit 34, and conduit 36 is fitted with valve 37 which is mechanically or electrically linked to the float 8. Depending on the level of the liquid in the filtering compartments this float ensures, by controlling the suction of pump 9, that the level of the liquid is sufficient to submerge the pre-filter and filter. Conduits 38 and 39, also equipped with valves, enable respectively the injection of the washing water under pressure and of the compressed air in order to carry out periodical counter flow cleaning of these filters. During the cleaning operation, the pump 9 is isolated by means of valve 40.

20 The washing water is recovered through the overflow 44, consisting of an inverted truncated cone with its upper edge above that of the weir 28. The washing water is discharged by pipe 12 and is eventually purified.

25 In the invention the separator operates as follows:

The mixture of the two non-miscible liquids (oil and water) arrives at the top of the separator via conduit 7, falls into the pre-separator whilst the oil is pre-separated and accumulates in the upper section defined by a horizontal surface at the level of the oil weir 14 and is at atmospheric pressure. As the accumulation of pre-separated oil proceeds, it is discharged over the edge and collected in compartment 15 where it is evacuated by gravity through pipe 17.

40 The pre-separated mixture then flows via the inlet collector 41 which is partitioned with radial baffles 43 activating the coalescence of the oil into the separator 42.

After going through the separator 42 the separated oil is gathered at the upper part of the separator 42 and then accumulates in the discharge chimney 25 whence it flows over the edge 26 into the collecting compartment 15. The cleaned water climbs through the annular chimney 27 and reaches the free surface at atmospheric pressure; the level of this free surface is determined by the overflow 28. The levels of the oil overflows 26 and 14 are higher than this reference level 28 so as to prevent water flowing into the collecting compartment 15, but still allowing an automatic discharge of the oil due to the density difference between water and oil.

60 After going over the overflow 28, the cleaned water successively passes through the pre-filter 29 and the filter 30 with an adequate flow thanks to the suction effect of the self-priming pump 9. The flow of this pump is controlled by the float 8 so that the prefilter and the filter are always sub-

merged. The small particles of oil still in suspension in the water are stopped by the oleophilic and coalescent actions of the filter bed. The cleaned water is discharged through the pump 9 and the pipe 35.

70 When the pressure of the suction circuit of pump 9 reaches a critical value indicating that the filter is clogged, the separation process stops and the filter is cleaned and washed. To do this, the arrival of polluted mixture is stopped, valve 40 is closed and valves in conduits 38 and 39 are opened letting in pressurised water and compressed air respectively. This mixture passes through the filter bed, at counter flow, going through the mushroom shaped sieves. Because of the free space above the filtering bed and the agitation created by the compressed air, the filtering media is placed in suspension and cleaned. The filter bed particles are retained by the prefilter, the washing water and the recovered oil going through the prefilter and discharged through the overflow 44 and re-enter the separation circuit of the mixture to be separated.

90 Between two separation processes, the separator remains full of liquid. The sediments which accumulate at the bottom of the separator are periodically discharged by means of valve 6.

95 The separator can, for instance, have several separating volumes side by side; the pre-filter could be fitted on its lower horizontal surface with pressurised water inlet pipes and compressed air inlet pipes so that it could be cleaned on its own. By removing the horizontal partition 20, the prefilter could be made into a sieve, of an adequate porosity, supported by or replacing the horizontal partition 21.

WHAT WE CLAIM:

1. A separator suitable for the separation of a first liquid and a second liquid having a higher specific gravity than that of the first liquid from a liquid mixture containing both liquids, the liquids being mutually immiscible, the separator comprising an inlet for introducing the mixture into a first separation zone so constructed and arranged as to promote the separation by gravity of a substantial part of the first liquid from the mixture, a first liquid outlet for the discharge of separated first liquid from the separator, a second liquid outlet for the discharge of separated second liquid from the separator, means for conducting first liquid separated in the first separation zone to the first liquid outlet, means for conducting the remaining liquid mixture containing a minor proportion of the first liquid and a major proportion of the second liquid towards the second liquid outlet along a flow path including a part extending downward through a second separation zone comprising a liquid-permeable bed of filter material



5 nected via valve 40 the self-priming suction pump 9 discharging the sucked water into conduit 35. Between the separator and the pump three conduits 36, 38 and 39 are connected to conduit 34, and conduit 36 is fitted with valve 37 which is mechanically or electrically linked to the float 8. Depending on the level of the liquid in the filtering compartments this float ensures, by controlling the suction of pump 9, that the level of the liquid is sufficient to submerge the pre-filter and filter. Conduits 38 and 39, also equipped with valves, enable respectively the injection of the washing water under pressure and of the compressed air in order to carry out periodical counter flow cleaning of these filters. During the cleaning operation, the pump 9 is isolated by means of valve 40.

20 The washing water is recovered through the overflow 44, consisting of an inverted truncated cone with its upper edge above that of the weir 28. The washing water is discharged by pipe 12 and is eventually purified.

In the invention the separator operates as follows:

30 The mixture of the two non-miscible liquids (oil and water) arrives at the top of the separator via conduit 7, falls into the pre-separator whilst the oil is pre-separated and accumulates in the upper section defined by a horizontal surface at the level of the oil weir 14 and is at atmospheric pressure. As the accumulation of pre-separated oil proceeds, it is discharged over the edge and collected in compartment 15 where it is evacuated by gravity through pipe 17.

40 The pre-separated mixture then flows via the inlet collector 41 which is partitioned with radial baffles 43 activating the coalescence of the oil into the separator 42.

45 After going through the separator 42 the separated oil is gathered at the upper part of the separator 42 and then accumulates in the discharge chimney 25 whence it flows over the edge 26 into the collecting compartment 15. The cleaned water climbs through the annular chimney 27 and reaches the free surface at atmospheric pressure; the level of this free surface is determined by the overflow 28. The levels of the oil overflows 26 and 14 are higher than this reference level 28 so as to prevent water flowing into the collecting compartment 15, but still allowing an automatic discharge of the oil due to the density difference between water and oil.

60 After going over the overflow 28, the cleaned water successively passes through the pre-filter 29 and the filter 30 with an adequate flow thanks to the suction effect of the self-priming pump 9. The flow of this pump is controlled by the float 8 so that the prefilter and the filter are always sub-

merged. The small particles of oil still in suspension in the water are stopped by the oleophilic and coalescent actions of the filter bed. The cleaned water is discharged through the pump 9 and the pipe 35.

70 When the pressure of the suction circuit of pump 9 reaches a critical value indicating that the filter is clogged, the separation process stops and the filter is cleaned and washed. To do this, the arrival of polluted mixture is stopped, valve 40 is closed and valves in conduits 38 and 39 are opened letting in pressurised water and compressed air respectively. This mixture passes through the filter bed, at counter flow, going through the mushroom shaped sieves. Because of the free space above the filtering bed and the agitation created by the compressed air, the filtering media is placed in suspension and cleaned. The filter bed particles are retained by the prefilter, the washing water and the recovered oil going through the prefilter and discharged through the overflow 44 and re-enter the separation circuit of the mixture to be separated.

90 Between two separation processes, the separator remains full of liquid. The sediments which accumulate at the bottom of the separator are periodically discharged by means of valve 6.

95 The separator can, for instance, have several separating volumes side by side; the pre-filter could be fitted on its lower horizontal surface with pressurised water inlet pipes and compressed air inlet pipes so that it could be cleaned on its own. By removing the horizontal partition 20, the prefilter could be made into a sieve, of an adequate porosity, supported by or replacing the horizontal partition 21.

WHAT WE CLAIM:

1. A separator suitable for the separation of a first liquid and a second liquid having a higher specific gravity than that of the first liquid from a liquid mixture containing both liquids, the liquids being mutually immiscible, the separator comprising an inlet for introducing the mixture into a first separation zone so constructed and arranged as to promote the separation by gravity of a substantial part of the first liquid from the mixture, a first liquid outlet for the discharge of separated first liquid from the separator, a second liquid outlet for the discharge of separated second liquid from the separator, means for conducting first liquid separated in the first separation zone to the first liquid outlet, means for conducting the remaining liquid mixture containing a minor proportion of the first liquid and a major proportion of the second liquid towards the second liquid outlet along a flow path including a part extending downward through a second separation zone comprising a liquid-permeable bed of filter material



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which is pulverulent or granular and which is active for retaining the coalescing droplets of the first liquid, means for causing a pressure reduction across the second separation zone so that liquid is drawn through the second separation zone under reduced pressure, and means for conducting the resulting liquid from the bottom of the liquid-permeable bed to the second liquid outlet.

2. A separator as claimed in claim 1 wherein a flow mechanism is provided to ensure that the liquid level is permanently above the filter bed in the second separation zone.

3. A separator as claimed in either of claims 1 and 2 wherein a pre-filter is provided above the bed of filtered material.

4. A separator as claimed in any one of the preceding claims which includes means for interrupting the separation of the mutually immiscible liquids and means for back-washing the filter bed by a counter flowing stream of the second liquid and air and means for returning the used back-washing second liquid to the inlet.

5. A separator suitable for the separation of oil from water comprising means for introducing an oil-water mixture into a first separation zone at atmospheric pressure wherein a substantial part of the oil can be separated by gravity from the water: means for removing this separated oil through a first liquid outlet: means for passing the remaining water which comprises a minor proportion of oil to a second separation zone where it is passed downward through a pre-filter and a filter bed comprising a filtration material which is pulverulent or granular and which is capable of filtering and coalescing droplets of oil, pumping means being provided for drawing the water towards a second liquid outlet through the

second separation zone under reduced pressure, together with means for removing and discharging the separated water through the second liquid outlet.

6. A separator according to any one of the preceding claims in which a vertical cylindrical envelope forms a tank having a dome-shaped upper part fitted with a mixture inlet pipe and a rod of a float; the lower part of this envelope in the shape of a truncated cone is fitted with a sediment drainpipe and outlet pipe for the cleaned second liquid; the envelope possesses internal partitionings which are fitted together to form a pre-separator and the first separation zone; and the envelope contains a pre-filter and the filter bed which operates under suction created by a pump incorporated in the second liquid outlet pipe.

7. A separator according to claim 1 substantially as hereinbefore described with reference to the drawings.

8. A method of separating oil from water comprising introducing the oil-water mixture into a separator according to any one of the preceding claims and obtaining the water substantially free of oil from the second liquid outlet.

9. A method as claimed in claim 8 in which a separator according to either of claims 4 and 7 is used wherein from time to time the separation is interrupted and a portion of washing water is used to back-wash the filter bed together with injection of air to clean the filter bed, the water used in the back-washing being returned for re-entry into the separator.

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