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(71) Applicant: OLIM TECHNOLOGIES SA 1005 Lausanne (CH)

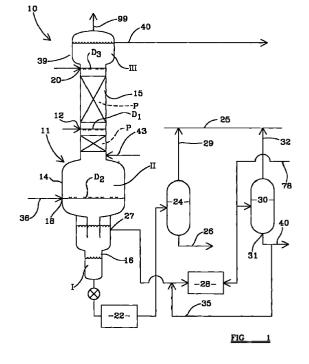
(72) Inventor: The inventor has agreed to waive his entitlement to designation.

(74) Representative:

Lucking, David John **FORRESTER & BOEHMERT** Franz-Joseph-Strasse 38 80801 München (DE)

## (54)Method of refining waste oil

(57)A method of refining waste oil containing contaminants and water comprising the steps of contacting the waste oil with an aliphatic solvent to form a waste oil/aliphatic solvent mixture, and in a treatment vessel (11) separating the mixture into three phases, namely a bottom phase (I) comprising predominantly water, an intermediate phase (II) comprising predominantly residuum containing contaminants and a top phase (III) comprising predominantly an oil/aliphatic solvent solution.



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## Description

**[0001]** This invention relates to a method of an apparatus for refining waste oil. Waste oil may contain many unwanted substances such as water, contaminants and additives which need to be removed before the oil can be re-used.

[0002] It is known to refine used or waste lubricating oil containing asphalt contaminants by mixing the waste oil with propane or the like such that oil is dissolved in the propane. The oil/propane solution is then separated from the residuum containing contaminants. For example in US Patent 2196989 there is described such a method in which contaminated oil is contacted in a continuous treatment tower, by an ascending stream of propane so that the contaminated oil becomes dissolved in the propane. A precipitant gas namely methane is introduced into the treatment tower and causes precipitation of already dissolved contaminated oily particles, so that these fall by gravity and are re-contacted by the propane further to remove the oil from the contaminants. This is described as refluxing and the result is said to be lubricating oil substantially free from asphalt contamination, and asphalt which is substantially lubricating oil free.

[0003] Waste or used oil however contains other unwanted substances particularly water. Thus the method described in US 2196989 cannot readily be adapted for the treatment of waste oil containing water. However in WO95/21902 of Mellen et al, there is described a method for the removal of contaminants from waste or used oil, in which a solvent, i.e. propane is used to dissolve the oil in a static mixer. In WO95/21902 however, if the waste oil contains too much water, this has to be removed before the waste oil is refined. Also, the water is removed from the static treatment vessel with residuum and then has to be separated therefrom.

**[0004]** Thus these known methods are not sufficiently efficient either at separating the oil/propane mixture from the contaminants and other residuum, or water, in a continuous process. Accordingly it is an object of the present invention to provide a new and improved waste oil refining method.

[0005] According to a first aspect of the invention we provide a method of refining waste oil containing contaminants and water comprising the steps of contacting the waste oil with an aliphatic solvent to form a waste oil/aliphatic solvent mixture, and in a treatment vessel separating the mixture into three phases, namely a bottom phase comprising predominantly water, an intermediate phase comprising predominantly residuum containing contaminants and a top phase comprising predominantly an oil/aliphatic solvent solution, and removing the three phases individually from the treatment vessel.

**[0006]** By separating the waste oil/aliphatic solvent mixture into three phases, a more efficient method is achieved, and it is not essential to reduce the amount of

water in the waste oil prior to refining as described in WO95/21902.

**[0007]** Preferably the waste oil and aliphatic solvent are mixed in the treatment vessel although they may be pre-mixed if desired and fed to the treatment vessel.

**[0008]** To enhance the separation of the phases as described, a precipitant gas such as nitrogen, carbon dioxide, or a  $C_1$  to  $C_4$  alkane may be introduced into the treatment vessel to cause refluxing of the waste oil/aliphatic solvent mixture in a similar manner to that described in US 2196989.

**[0009]** Alternatively another refluxing agent could be used, such as for example only, recovered oil from downstream in the refining process. In each case a continuous treatment method can be carried out compared with the static method of WO95/21902. Preferably, to further facilitate such refluxing, the treatment vessel has a larger cross section in a lower region thereof than in an intermediate region above the lower region, refluxing occurring above the lower region and the bottom and intermediate phases collecting in the lower region of the treatment vessel.

[0010] The precipitant gas or other refluxing agent conveniently is introduced into an upper part of the intermediate region of the vessel and the waste oil is introduced below into the treatment vessel for mixing with the aliphatic solvent. The aliphatic solvent is preferably introduced into the treatment vessel in a lower part of the intermediate region thereof, preferably below the waste oil.

[0011] Whereas in order to achieve a useable recovered oil, necessarily one has to separate the oil from the aliphatic solvent, each of the three phases may contain some entrained aliphatic solvent and so desirably each of the three phases is treated to remove aliphatic solvent therefrom. The recovered aliphatic solvent is preferably recycled by mixing with further waste oil in the treatment vessel.

**[0012]** When the recovered oil has been cleaned of aliphatic solvent, preferably the oil from the top phase is subjected to a fractional distillation e.g. vacuum distillation to separate the oil into different fractions.

[0013] According to a second aspect of the invention we provide an apparatus for performing the method of the first aspect of the invention, comprising a treatment vessel in which a waste oil/aliphatic solvent mixture is separated into three phases, namely a bottom phase comprising predominantly water, an intermediate phase comprising contaminant residuum and a top phase comprising and oil/aliphatic solvent solution, the treatment vessel having means to enable each of the three phases to be removed individually from the treatment vessel.

**[0014]** The treatment vessel may comprise a lower region having a greater cross section that an intermediate region above the lower region to enable refluxing of the waste oil/aliphatic solvent mixture in the treatment vessel above the lower region and to aid separation of

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the three phases.

[0015] The apparatus may comprise an evaporator means for evaporating from the bottom phase and/or top phase and/or intermediate phase, aliphatic solvent and may include a fractional distillation means for separating the oil from the top phase into different fractions.

[0016] The oil may be vaporised prior to distillation in an evaporator means comprising a vessel having passage means along which oil to be evaporated, passes as the oil is heated, the passage means being lined with a flowing film of heavy fraction obtained from the fractional distillation means.

[0017] According to a third aspect of the invention we provide an evaporating means for vaporising oil, the evaporator means comprising passage means along which the oil passes as the oil is heated, the passage means being lined with a flowing film of heavy fraction oil.

[0018] The invention will now be described with the aid of the accompanying drawings in which:-

FIGURE 1 is a diagram of a first part of an apparatus for performing the invention;

FIGURE 2 is a diagram of a second part of the apparatus.

[0019] Referring to the drawings an apparatus 10 for refining waste oil containing contaminants including water, comprises a treatment vessel 11 parts of which are heated to a temperature of between 15° and 45°C, and into which the waste oil is introduced at point 12 from a storage vessel or the like along a disperser D1, preferably at a temperature in the range 15°C and 45°C. The treatment vessel 11 comprises a tower having a lower region 14 the cross section of which is generally larger than an intermediate region 15 immediately above. At the very bottom of the lower region 14 there is a sump 16 for a purpose hereinafter explained. Point 12 is preferably in the intermediate region 15.

[0020] In this example, the waste oil is contacted in the treatment vessel 11 with an aliphatic solvent being propane liquid, although in another example, the waste oil and propane or other aliphatic solvent may be contacted in a separate mixing vessel before being introduced into the treatment vessel 11.

[0021] The propane is introduced into the treatment vessel 11 at point 18 at a lower part of the intermediate region 15, via a disperser D2 from a line 36 from a propane store (not shown). Desirably, the propane is heated or cooled before being introduced into the treatment vessel 11 to be at a temperature of between -5°C and 45°C.

[0022] It will be appreciated that the propane and waste oil will form a mixture with a large proportion of the waste oil dissolved in the propane and water will tend to sink in the vessel 11 by virtue of its higher specific gravity. The ratio of propane to waste oil in the mixture in the vessel 11 is preferably in the range 3:1 to

10:1.

[0023] Insoluble contaminants, additives and water with some bound oil will fall by gravity in the treatment vessel 11 towards the lower region 14 in small droplets which will contact the incoming propane which will remove some of the bound oil from the contaminants. A precipitant gas such as nitrogen is injected into the treatment vessel 11 preferably at a temperature of between 15°C and 45°C at point 20 being an upper part of the intermediate region 15, via a disperser D3. This becomes dissolved in the propane/oil solution, and as nitrogen concentration increases and due to the low solubility of the dissolved oil in nitrogen, some of the contaminated waste oil originally dissolved in the propane will be precipitated and fall by gravity towards the lower region 14 of the vessel 11. While falling, the waste oil will contact countercurrently the ascending solution of clean propane and dissolved oil so that more of the oil will be dissolved thus separating more oil from contaminants.

[0025] Due to this effect and the inflowing waste oil at point 12, refluxing occurs in the intermediate region 15 of the treatment vessel 11 enhancing the efficiency of the waste oil cleaning process. As a result, the waste oil/propane mixture separates into three phases, namely a bottom phase I which is predominantly water with some entrained propane and other contaminants, an intermediate phase II which is predominantly waste residuum with some entrained propane and water, and a top phase III which is an oil/propane solution which collects in an upper region 39 of the treatment vessel 11. The upper region 39 has a cross section greater than that of the immediately adjacent part of the intermediate region 15, but less than that of the lower region 14.

[0026] The bottom phase I tends to collect in the very bottom of the lower region 14 of the treatment vessel 11, in sump 16 from where the bottom phase may be removed and fed to a heating vessel 22 and hence to an evaporating means 24 which comprises in this example a flash evaporator. There any entrained propane is removed and delivered via a line 29 to a collection line 25 from where the propane may be removed, cleaned and recycled to the treatment vessel 11. The water is removed from the flash evaporator 24 via a line 26 for disposal.

[0027] The intermediate phase II tends to collect in the lower region 14 of the treatment vessel 11 too, and is removed via a poll 27, fed to a heating vessel 28 and hence to an atmospheric pressure propane stripper 30 where the intermediate phase is further heated to remove propane which is fed to collection line 25 via a line 32 via another heater and a flash condensate receiver so that any waste water entrained in the propane is removed. The remaining residuum is removed from the stripper 30 via a port 31 and may be used for example only, when mixed with oil, as a road making material. Such oil may be any oil having a flash point

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above 200°C such as for example oil from downstream in the process, from line 74 (see below).

[0028] It can be seen that a portion of the residuum is circulated via line 35 back through the heating vessel 28 and again into the atmospheric propane stripper 30, to 5 aid the efficiency of the propane removing process.

The top phase III is removed from the upper region 39 of the treatment vessel 11 and fed along a line 40 to a surge vessel 41 (figure 2), so that any residuum entrained in the oil/propane solution can be removed and returned to the treatment vessel 11 along a return line 43 for further processing. The residuum is preferably at a temperature of between 15°C and 45°C. Any propane removed from the solution in the surge vessel 41 is fed via line 34 to collection line 25 for further treatment and recycling. The remaining cleaned top phase is then fed along a line 45 to a series of heaters 46-48 where the temperature of the solution is steadily raised. The hot solution is then fed along line 50 via a final heater 51, and into solvent flash column 53 where the major portion of the propane in the solution is removed, condensed in condensers 54,55 and recycled to the treatment vessel 11 via a line 36.

[0030] Remaining propane in the top phase solution is removed in a second (steam) stripper 58, and passes from the stripper 58 to a heater 60 and condenser 61 so that any light hydrocarbon fractions removed from the oil in the second stripper 58 can be recovered and fed to a store along line 62 for use. Any water in the propane arising from steam introduced into the second stripper 58 at 59 is also condensed in condenser 61 and may be collected in a sump 65 thereof for disposal.

[0031] The oil separated from the propane in the second stripper 58 is then treated by fractional distillation in a vacuum distillation column 70 from where the various fractions in the oil e.g. (VGO) gas oil 66, (light) neutral oil 67 and (heavy) neutral fuel oil 68 can be separated as is well known in the art.

[0032] Prior to introduction of the oil into the distillation column 70, the oil is preheated in a heat exchanger 71. Conventionally immediately prior to introduction into a distillation column, oil is vaporised by reducing the pressure of the oil. The pressure of the oil is however maintained in the heat exchanger 71 by means of the operation of a control valve C so that in the event of any remaining contaminants in the oil, these are not deposited in the heat exchanger 71 if the lighter oil fractions boil to dryness, which could cause a blockage and hence reduction in oil flow.

**[0033]** The hot oil from heat exchanger 71 is however vaporised in a falling film evaporator indicated at 72, prior to introduction into the distillation column 70 at line 76.

**[0034]** Any light fractions in the hot oil from heat exchanger 71 flash in a vessel 75 and by pass the evaporator 72 via a line 77 from where they are fed into line 76 to enter the distillation column 70.

[0035] The falling film evaporator 72 comprises a plu-

rality of tubular passages 73 through which the oil passes as it is heated. To avoid the possibility of contaminants being deposited in the passages, the passages are lined with a flowing film of oil. The oil for the film is obtained in this example from the distillation column 70 and comprises the heaviest fraction separated from the oil, known as fuel oil, which is fed to the falling film evaporator 72 via line 74 and the flash vessel 75 to which the hot oil from the heater 71 is also directed.

**[0036]** However the oil for the flowing film may be obtained from another source if desired.

[0037] If desired at least a proportion of the heat in the fraction oil from line 74 may be introduced into the pressure propane stripper 30 via a line 78, in order to maintain constant the level of distillation bottoms in the system. Alternatively, the line 78 may lead to a store.

[0038] Preferably the volume of oil flowing to provide the falling film is substantially greater, typically ten times greater than the volume of the recovered oil being heated so that an insubstantial amount of the oil of the falling film only, is evaporated, and the flowing film is relatively thin (1 mm or so) compared with the volume of the passages 73 (30-35mm diameter perhaps) lined by the flowing film. The falling film effectively covers the passage walls but fills less than 10% of the passage void, more preferably 6%. Thus the recovered oil can efficiently be heated in the evaporator 72 to evaporate the oil, without risk of any contaminants remaining in the recovered oil, being deposited.

[0039] The falling film evaporator 72 may be used in other applications to that described herein, for example in any application where it is desired to evaporate oil. For example, a falling film evaporator 72 may be used in the place of any of the evaporating and flash vessels described above in relation to the oil refining process.

**[0040]** Various modifications may be made without departing from the scope of the invention.

[0041] For example the treatment vessel 11 shown in the drawings is only an example of a suitable vessel in which a waste oil/propane mixture may be separated into three distinct phases. Although it is preferred that propane or other aliphatic solvent removed from the three phases is collected, treated and recycled, virgin propane or any other suitable aliphatic solvent may be used. The number of and positions of heaters used in the apparatus 10 may be varied depending on the refinery layout and the positioning of each of the various vessels may be arranged to suit a particular site.

[0042] As described, the treatment vessel 11 is heated to a temperature of between 15°C and 45°C by virtue of the flows into the vessel, at 12, 20 and from line 43, but ancillary heaters/coolers may be provided if desired.

[0043] Although as described nitrogen is introduced into the treatment vessel to facilitate refluxing, in another example another precipitating/refluxing agent, such as carbon dioxide, or a C1 to C4 alkane gas may be used. In any event, such gas is introduced into treat-

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ment vessel 11 at pressure, typically a pressure of less than one bar above the pressure at which the propane liquid is introduced into the vessel 11. The nitrogen or other gas is removed from the top vessel 11 via line 99 and recovered for re-use or is mixed with combustion air for use in heating in the apparatus. If desired other substances which facilitate precipitation of the oil and contaminants may be introduced into the treatment vessel 11 in addition to or instead of the precipitant gas, such as flocculating reagents.

**[0044]** Further alternatively, a refluxing agent being distillation feed oil from downstream in the process, may be used. Such refluxing oil may be fed from between the stripper 58 and the heating vessel 71, as indicated in dotted lines in figure 2.

**[0045]** If desired, the treatment vessel 11 may be at least partially packed as shown at P to facilitate mixing of the waste oil and propane and the effect of the refluxing agent, as well as settling out of the phases.

**[0046]** The features disclosed in the foregoing description, or the following claims, or the accompanying drawings, expressed in their specific forms or in terms of a means for performing the disclosed function, or a method or process for attaining the disclosed result, as appropriate, may, separately, or in any combination of such features, be utilised for realising the invention in diverse forms thereof.

## Claims

- 1. A method of refining waste oil containing contaminants and water including the steps of contacting the waste oil with an aliphatic solvent to form a waste oil/aliphatic solvent mixture, and in a treatment vessel (11) separating the mixture into three phases, namely a bottom phase (I) including predominantly water, an intermediate phase (II) including predominantly residuum containing contaminants and a top phase (III) including predominantly an oil/aliphatic solvent solution, and removing the three phases (I, II, III) individually from the treatment vessel (10).
- 2. A method according to claim 1 characterised in that the waste oil and aliphatic solvent are mixed in the treatment vessel (10).
- A method according to claim 1 or claim 2 characterised in that refluxing agent comprising a precipitant gas is introduced into the treatment vessel (10) to cause refluxing of the waste oil/aliphatic solvent mixture.
- 4. A method according to claim 1 or claim 2 characterised in that a refluxing agent being recovered oil from downstream in the refining process, is introduced into the treatment vessel (11) to cause refluxing of the waste oil/aliphatic solvent mixture.

- 5. A method according to claim 3 or claim 4 characterised in that the treatment vessel (11) has a larger cross section in a lower region (14) thereof than in an intermediate region (15) above the lower region (14), refluxing occurring above the lower region (14) and the bottom (I) and intermediate (II) phases collecting in the lower region (14) of the treatment vessel (11).
- 6. A method according to claim 5 characterised in that the refluxing agent is introduced into an upper part of the intermediate region (15) of the vessel (11), and the waste oil below.
- 7. A method according to any one of the preceding claims characterised in that the aliphatic solvent is introduced into the treatment vessel (11) in a lower part of the intermediate region (15) thereof.
- 20 8. A method according to any one of the preceding claims characterised in that each of the three phases (I, II, III) is treated to remove aliphatic solvent therefrom, and the recovered aliphatic solvent is recycled by mixing with the waste oil.
  - 9. A method according to any one of the preceding claims characterised in that the oil from the top phase (III) is subjected to a fractional distillation to separate the oil into different fractions.
  - 10. An apparatus (10) for performing the method of any one of the preceding claims including a treatment vessel (11) in which a waste oil/aliphatic solvent mixture is separated into three phases, namely a bottom phase (I) including predominantly water, an intermediate phase (II) including residuum containing contaminants and a top phase (III) including an oil/aliphatic solvent solution, the treatment vessel (11) having means to enable each of the three phases to be removed individually from the treatment vessel (11).
  - 11. An apparatus (10) according to claim 10 characterised in that evaporator means (22, 28) are provided for evaporating from the bottom phase (I) and/or top phase (III) and/or intermediate phase (II), aliphatic solvent.
  - 12. An apparatus (10) according to claim 10 or claim 11 including a fractional distillation means (70) for separating the oil from the top phase (III) into different fractions.
  - 13. An apparatus according to claim 12 wherein the oil is vaporised prior to distillation in an evaporator means (72) including a vessel having passage means (73) along which oil to be evaporated, passes as the oil is heated, the passage means

(73) being lined with a flowing film of heavy fraction obtained from the fractional distillation means (70).

**14.** An evaporating means (72) for vaporising oil, the evaporator means (72) comprising passage means (73) along which the oil passes as the oil is heated, the passage means (73) being lined with a flowing film of heavy fraction oil.

