Feb. 12, 1952 C. W. STRATFORD 2,585,874

TREATING LUBRICATING DISTILLATES

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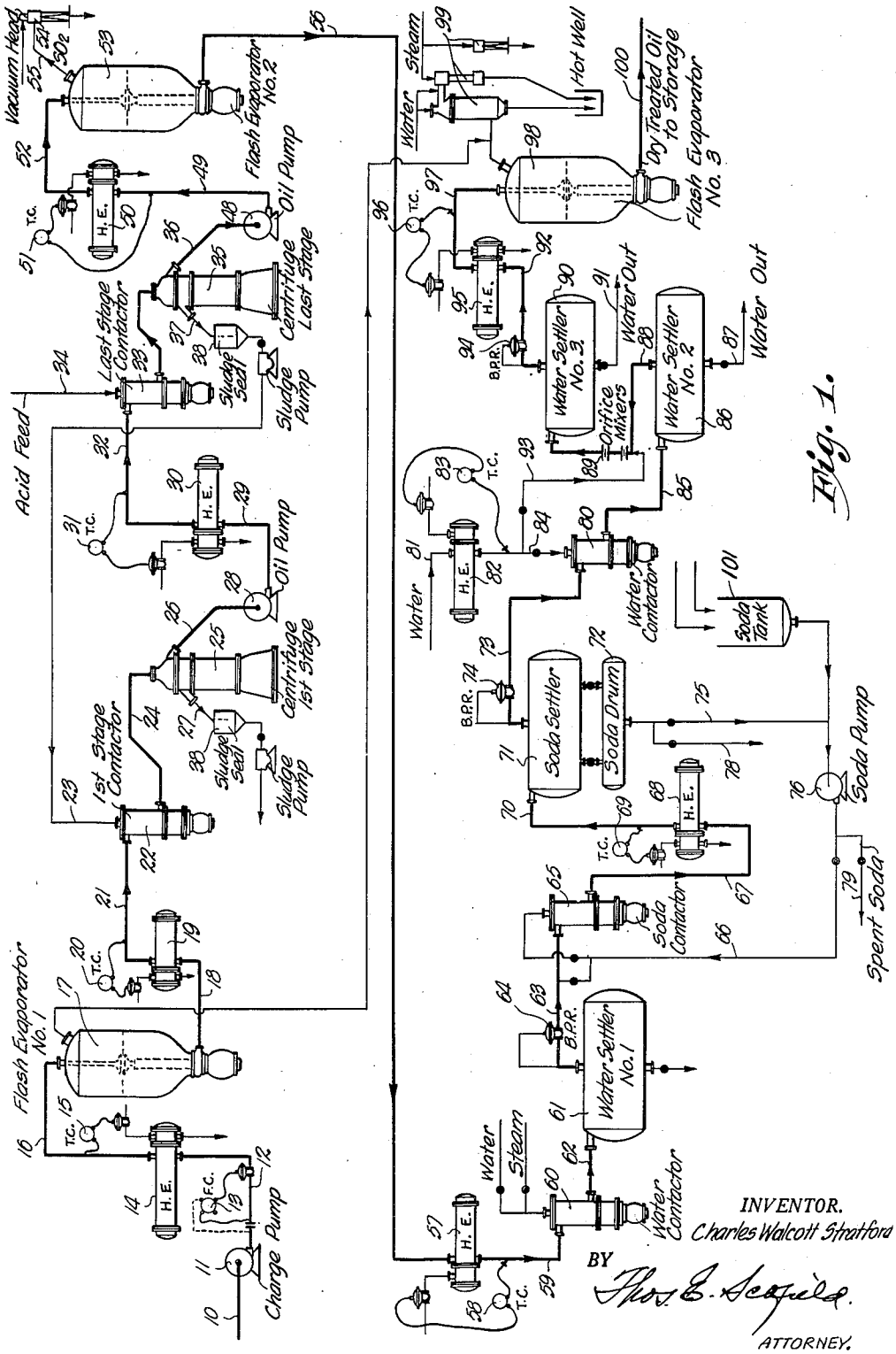


Fig. 1.

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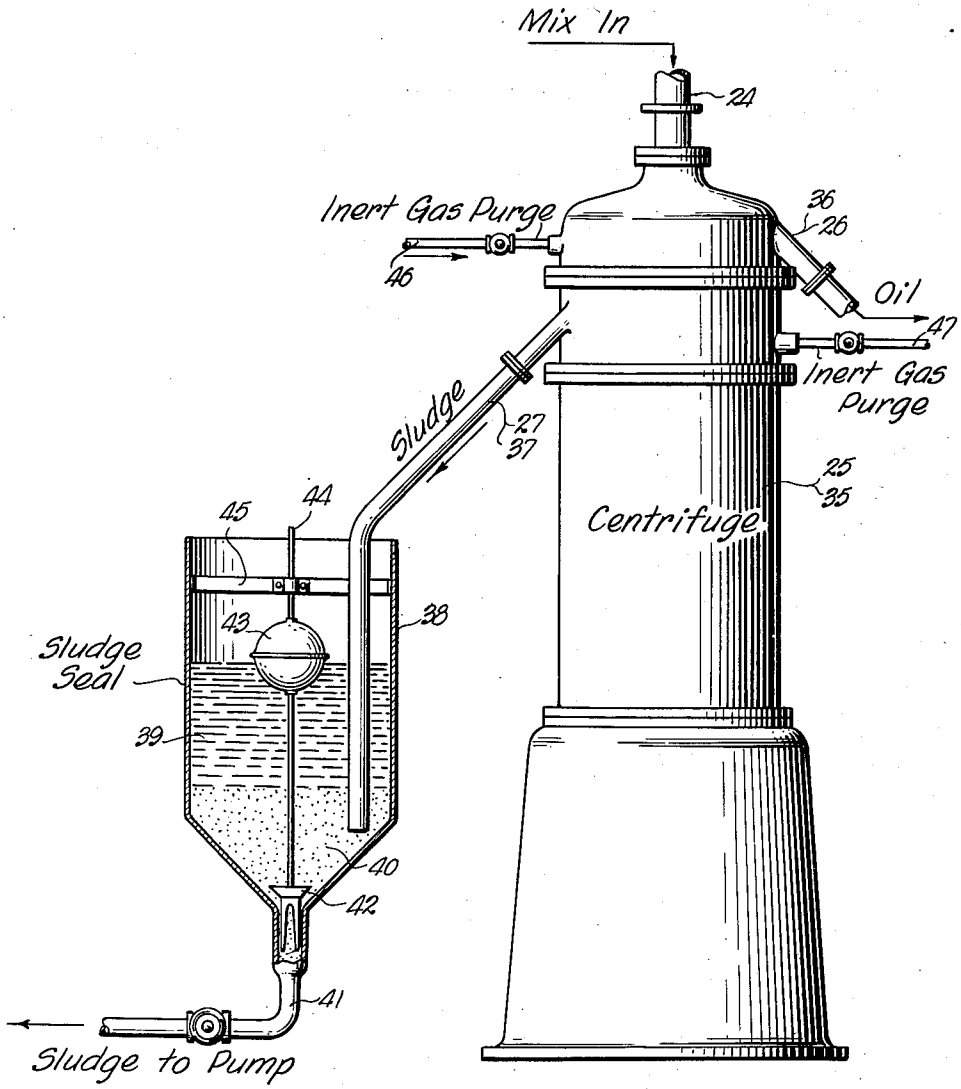


Fig. 2.

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TREATING LUBRICATING DISTILLATES

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4 Claims. (Cl. 196—38)

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This invention relates to improvements in treating lubricating oil distillates and refers more particularly to an acid treating method in which the oil is preliminarily dehydrated and thereafter contacted with the acid in successive steps at a predetermined optimum elevated temperature for the particular oil undergoing treatment, after which sulphur dioxide and sulphonic compounds are removed and the acid-treated oil water washed and subjected to soda or caustic treatment at elevated temperatures and pressure.

The novelty resides primarily in complete elimination of air from the system during treating, the removal of sulphur dioxide, volatile sulphur compounds and finely dispersed sludge particles and sulphonic bodies between the acid and neutralization steps and the neutralization and water washing of the treated oils under conditions of elevated temperature and pressure.

The method is an improvement of a prior Patent No. 1,927,853, issued September 26, 1933. Treating, as described in this patent, is performed in substantially a closed system, yet it has been found that even the air which is normally absorbed in hydrocarbon distillates in tankage and the air which is present in centrifuge heads contains sufficient oxygen to have a deleterious effect on the color of the finished product and the fluidity of the sludge produced. This is particularly true of the air which contacts the oil and sludge within the centrifuge heads, since they cannot be operated under pressure and the oil and acid discharged from the rotating elements are thrown in a high velocity stream through oxygen containing atmosphere. The invention contemplates, therefore, not only complete deaeration of the feed stock but also blanketing of the centrifuge heads with inert gas and the sealing of sludge receivers under a cover of acid neutral oil.

It has been found that many oils treated by the method described in my aforementioned patent are difficult to neutralize and wash in the conventional manner with caustic soda and water because of the formation of emulsions during neutralization and water washing, which emulsions are extremely difficult to break and settle. In many cases it has been found impossible to neutralize the acid-treated oils with caustic and, as a consequence, other methods had to be resorted to such as clay contacting and filtration. This difficulty with emulsions can be minimized and to a great extent eliminated by the rapid and complete removal of sulphur dioxide vapors

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from the oil immediately following the last acid treating stage, followed by a quick contacting step with a small quantity of hot water or steam. A rapid moisture treatment for a short period at this point serves to coagulate the extremely minute practically invisible particles of sludge not separated in the centrifuge of the last acid treating step and this agglomerated sludge can be readily settled out together with some of the sulfonates formed during the acid treatment. When this quick water washing step is used the distillates can be satisfactorily and completely neutralized with caustic and settled without encountering difficulty from troublesome emulsions if conditions of elevated temperatures and pressures are maintained during subsequent neutralization and settling. This suggested operation not only results in reducing oil losses to a minimum but also greatly reduces the quantity of caustic necessary to effect neutralization.

In the accompanying drawings which form a part of the specification and are to be read in conjunction therewith and in which there is shown an embodiment of the invention,

Fig. 1 is a flow diagram of an apparatus adaptable to the method,

Fig. 2 is an enlarged detail showing the arrangement at the centrifuges for eliminating air from the centrifuge discharge streams.

Referring to the drawings and particularly Fig. 1, the apparatus will be described by following the steps of a typical operation. Lubricating oil distillate is supplied from any convenient source through pipe 10 and is picked up by charging pump 11 through which it is supplied to the system through line 12 at a uniform rate by flow control device 13. The oil passes first through heat exchange unit 14 where it is heated by steam or other suitable heating medium to a temperature sufficiently high to permit evaporation of the moisture absorbed or entrained therein, the temperature being held constant by means of temperature control device 15 known to the art. The oil at this elevated temperature is then passed through pipe 16 to flash evaporator 17 where it is stripped of whatever air and moisture it contains under reduced pressure. This flash evaporator is preferably of the type shown in my Patent 2,368,049 dated January 23, 1945, or it may be of any other suitable type, such as a spray or baffle tower, conventionally used for like purposes. On discharge from the evaporator through pipe 18 the oil is again passed through a heat exchange unit 19 where it is heated or cooled to the temperature best suited to the treatment of the par-

ticular oil. Depending upon the lubricating oil being treated, this temperature will normally be within the range of from 100° F. to 275° F. The oil temperature is governed as before by temperature control device 20 interposed in line 21 through which the oil is discharged from the heat exchanger which regulates a valve controlling the amount of heating or cooling medium circulated through the exchanger 19. The temperature of the oil passing to the first acid contact step and to subsequent acid treating steps may be as low as 100° F. and as high as 300° F. In the first stage contactor 22 the oil is intimately intermixed with acid supplied through pipe 23. From the contactor the mixture of oil and acid passes through pipe 24 to a centrifuge separator 25 where the oil and acid sludge are separated, the former discharged through pipe 26 and the latter through pipe 27. The oil separated in the first stage centrifuge is picked up by pump 28 and is directed through pipe 29 to heat exchanger 30 where the temperature of the oil is again brought to an optimum for the subsequent treating step by temperature control device 31. From the heat exchanger the oil passes through pipe 32 to a second or last stage contactor 33 into which acid is supplied through pipe 34. Here, as in the first stage, the acid and oil are intimately contacted and the mixture passed to the second or last stage centrifuge 35. The separating operation in this stage corresponds to that in centrifuge 25, the oil passing off through pipe 36 and the acid sludge through pipe 37. From both centrifuges 25 and 35 the sludge withdrawal is performed through an apparatus as shown in Fig. 2. It will be noted that the acid sludge leaving the centrifuge of each of these stages passes first to a sludge seal before entering the sludge pump. A cross section of the sludge seal reveals that it comprises a vessel 38 in which a body of oil 39, inert to the acid shown at 40, is maintained to float upon the surface of the acid sludge. Provision is made for limiting the flow of sludge to the sludge pump which comprises a float-operated plug 42 mounted as a valve in the sludge outlet line from the receiver or vessel 38. The float 43 and plug are mounted on a rod 44 slidably mounted in guides or supports 45 within the vessel. The floats rises upon the upper surface of the oil and follows the oil level to either seat or unseat the plug 42 in the conical bottom outlet of the vessel. When the sludge level is low the float drops and throttles the flow of sludge to the pump. When the level of the sludge rises the plug valve is lifted from its seat and an increased amount of sludge is discharged to the pump. Fig. 2 also indicates the inert gas purge lines 46 and 47 used to displace the air from the centrifuge heads providing an inert medium through which the streams issuing from the centrifuge bowl are discharged. It will be understood that means other than the one described and shown in Fig. 2 may be used for eliminating the contact of air with the sludge discharged from the centrifuges. For example, the sludge seal or vessel may be closed at the top, the oil layer omitted and a purge of nitrogen or other inert gas introduced above the sludge level, as is shown in the top of the centrifuge. It will also be understood that, although not shown, it is contemplated that the inert gas purge will be used on the heads of both of the centrifuges 25 and 35 in the apparatus shown in Fig. 1.

The acid treated oil leaving the centrifuge of the last stage contains a considerable quantity of sulphur dioxide (SO_2) vapors, both mechanically entrained and absorbed, the amount of these vapors generally increasing with increased acid applications. To remove these vapors in order to reduce subsequent caustic consumption and facilitate settling, the sour oil is forced by pump 48 and pipe 49 through heat exchanger 50 where the temperature is brought to that necessary for reducing the oil to a point where it can be readily atomized and where the sulphur dioxide will pass off as vapor under conditions of reduced pressure. Temperature control device 51 controls the temperature of the oil discharged from the heat exchanger within this optimum range. Passing from the heat exchanger the heated oil flows through line 52 into flash evaporator 53 which is preferably of the same type described previously and identified by the numeral 17 in the drawing. A vacuum is maintained in evaporator 53 by conventional steam ejector device 54 connected to the top of the evaporator through line 55. The SO_2 vapors and other volatile malodorous gases formed during treatment are removed from the system through line 55 connected into the top of the evaporator.

The sour oil from the flash evaporator still contains some small traces of acid sludge in the form of finely dispersed invisible particles, which escape separation even in the most efficient centrifuges, as well as certain sulfonic bodies. In order to remove these deleterious materials which are removable with water, the sour oil is drawn off from the bottom of the evaporator and is directed through pipe 56 to heat exchanger 57 where the temperature is brought within a range of from 150° F. to 300° F. by temperature control mechanism 58. The heated oil then passes through pipe 59 to water contactor 60 where it is intimately mixed with hot water and the mixture directed to water settler 61 through drawoff pipe 62. In the settler the mixture is separated into an oil layer and a water layer, the latter containing the separated sludge and/or sulfonic bodies. This separation is quite critical to the quantity of water used and the temperature at which the mixing takes place. It has been found that very small quantities of water will satisfactorily remove these undesirable components, whereas larger quantities of water will introduce difficulties in the settling operation. For most oils the quantity of water to be used will be in the order of one-tenth of one per cent to one percent by liquid volume of the oil although as high as five per cent is satisfactory for other oils. Temperatures normally employed should be 150° F. to 300° F. which necessitates holding an appreciable back pressure on the contacting and settling system to avoid vaporization of water. Pressures suitable for imposition on the water treatment step will vary from 10 pounds to 100 pounds per square inch.

The oil leaving the water settler is drawn off from the top through pipe 63 in which is interposed back pressure regulating valve 64. From the settler the oil is passed next to a soda contactor 65 where it is intimately mixed under the same conditions of elevated temperature and pressure with a caustic soda solution or other neutralizing agents supplied through pipe 66. Upon being discharged from the contactor through pipe 67, the oil-soda mixture again passes through a heat exchanger 68 where the temperature of the liquid is regulated to an opti-

mum by temperature control device 69. After passing through heat exchanger 68 the mixture is directed through line 70 to soda settler 71 where the oil and soda mixture is permitted to separate, the soda being drawn off and accumulated in soda drum 72, while the soda treated oil is removed from the top through pipe 73. Interposed in line 73 is a back pressure regulator 74. In some cases it is necessary to maintain a different temperature in the soda settling step than in the preliminary water settling step 61 in order to reduce the settling time required for breaking out the caustic solution from the oil. For this reason heat exchanger 68 is provided ahead of the soda settler so the mixture of oil and caustic can be heated or cooled to the desired optimum temperature prior to settling.

The settled soda solution accumulated in the soda drum 72 may be either recycled through pipe 75, pump 76 and pipe 66 to the soda contactor 65 or discharged from the system through pipes 78 or 79. A soda tank 101 is provided for blending the soda and water used in this step.

When neutralization is done under elevated temperatures and pressures, as indicated, the optimum strength of the neutralizing solution may be very low and, in the case of caustic soda, best results are usually obtained when neutralizing with 3% to 10% solutions. After neutralization the oil is passed to a final water contactor 80 which is also operated under conditions of elevated temperature and pressure. The water for washing is supplied through pipe 81 and is heated either by indirect heat exchange, as shown at 82, regulated by temperature control device 83, or by direct injection of steam into the water. The heated water is supplied to the contactor through pipe 84. The oil leaving the soda settler is thoroughly mixed with the hot water in the contactor. The mixture of oil and water passed from the contactor 80 through pipe 85 to water settler 86 from which water is drawn off at 87 and the oil through pipe 88. In the line 88 are orifice mixers 89 or other contacting device such as a duplicate of contactor 80 through which the oil passes upon being discharged from water settler No. 2, designated by reference numeral 86. It is passed thence to a second water settler 90, No. 3 in the drawing, where additional water is removed through pipe 91, the oil from which a substantial part of the water has been separated passing off from the top through line 92. Between lines 84 and 88 is provided a valve controlled by-pass line 93. In order to prevent vaporization of water within this water washing and settling steps, a back pressure, usually in the order of 10 pounds to 100 pounds per square inch, is maintained by back pressure regulator 94. The quantity of water used in this step will normally vary from 5% to 100% by volume of the oil being washed. This final water washing may be accomplished in one stage although it has been preferable to utilize two stages of mixing and settling in most cases.

The neutralized oil leaving the final water settler contains some water held in the oil either in an absorbed condition or by mechanical entrainment giving it a cloudy appearance at normal temperatures. In order to remove this remaining moisture and finish the oil to brightness, it is again heated in heat exchanger 95 whose temperature is regulated by control 96 and is then passed through pipe 97 to a third flash evaporator 98 similar to the other two previously described where it is flashed under conditions of elevated temperature and reduced pressure. A vacuum is maintained on the flash evaporator by means of a barometric condenser and steam condensing head, diagrammatically shown at 99, or by a vacuum pump or other conventional means. The heat exchanger 95 provided in the line between the water settler and flash evaporator brings the oil to the temperature required for dehydration, which is normally within the range of 165° F. to 210° F. depending upon the boiling range and viscosity of the oil being dehydrated. The dehydrated finish oil is pumped away from the flash evaporator through pipe 100 and coolers, not shown, to storage.

Thus, it will be seen that there has been provided a method by which lubricating oil distillates are acid treated within a temperature range which is accurately regulated to maintain optimum conditions for treating. While two steps of acid treating have been shown, it is contemplated that additional steps may be used according to the requirements of the particular oil and the acid concentration used in the respective steps. There has also been provided an improved method by which dispersed sludge, sulfonic bodies and objectionable components are separated by a water washing step interposed between the acid treatment and neutralizaton. The conditions of acid treatment are regulated to maintain them within the critical range required to eliminate the deleterious substances from the sour oil prior to soda treating. Finally, there has been supplied a neutralization and final water washing under accurately controlled conditions of temperature and pressure making for a more complete and efficient operation and considerably reducing losses common to previous methods.

Thus, it will be seen that there has been provided a method well adapted to attain the ends and objects hereinbefore set forth together with such other advantages which are obvious and which are inherent to the process. It will be understood that certain features and subcombinations are of utility and may be employed without reference to other features and subcombinations. This is contemplated by and is within the scope of the claims.

As many possible embodiments may be made of the invention without departing from the scope thereof, it is to be understood that all matter herein set forth or shown in the drawings is to be interpreted as illustrative and not in a limiting sense.

Having thus described my invention, I claim:

1. A method of treating lubricating distillates comprising the steps of heating the oil, dehydrating and deaerating it in an initial step, subjecting the oil to acid treatment in a plurality of steps with intermediate steps of acid oil separation, removing volatile sulfur compounds in the vapor phase from the acid treated oil in a stage of reduced pressure after acid treatment, next intimately mixing the treated oil with water, removing moisture from the oil, and then subjecting it to final steps of neutralization and water washing.

2. A method as in claim 1 wherein the temperature of the oil is separately controlled prior to its introduction to each step of acid treatment and wherein both the temperature and pressure on the water washing and neutralization steps is separately controlled.

3. A method as in claim 1 wherein the oil being treated is at all times kept from contact with the oxygen of the atmosphere prior to subjecting it

to the step of neutralization and wherein the acid sludge separated in each stage is at all times kept from contact with the oxygen of the atmosphere prior to its final discharge from the acid treating steps.

4. A method of treating lubricating distillates comprising the steps of heating the oil, dehydrating and deaerating it in an initial step, subjecting the oil to acid treatment in a plurality of steps with intermediate steps of acid-oil separation, removing volatile sulfur compounds in the vapor phase from the acid treated oil in a stage of reduced pressure, then intimately mixing steam with the treated oil, separating the moisture from the oil and subjecting it to water washing and neutralization and to final settling and evaporating steps to remove moisture and neutralizing materials.

CHARLES WALCOTT STRATFORD.

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