Oct. 15, 1929. P. M. TRAVIS 1,732,144

PROCESS OF REMOVING AMORPHOUS WAX FROM PETROLEUM OILS

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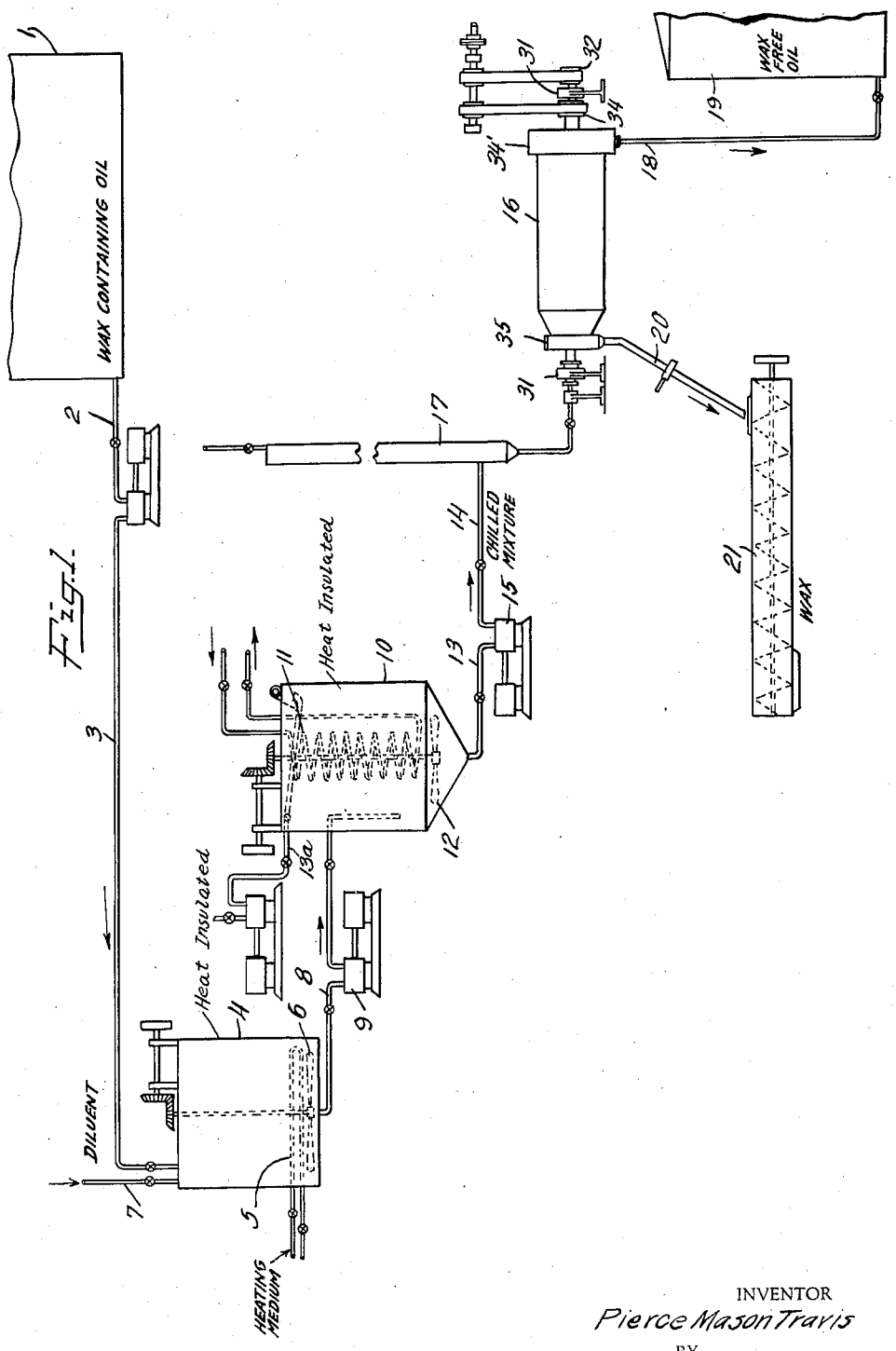


Fig. 1.

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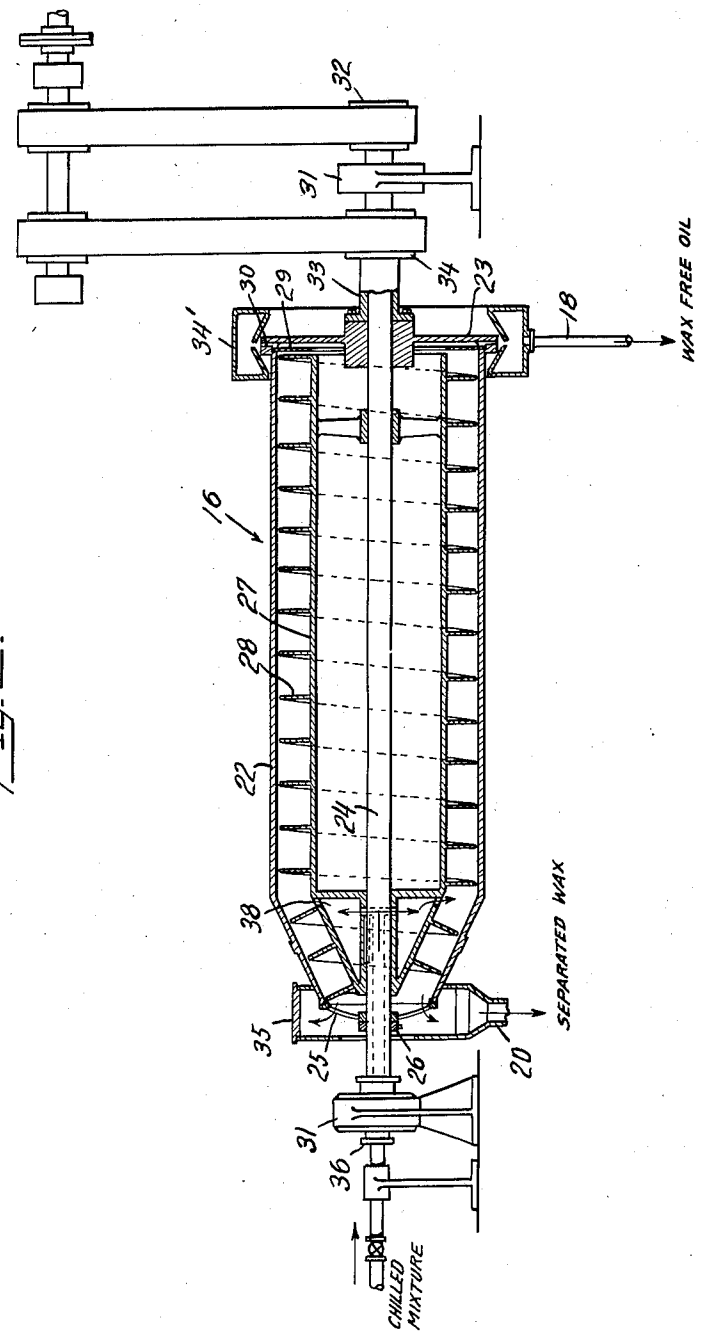


Fig. 2.

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UNITED STATES PATENT OFFICE

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PROCESS OF REMOVING AMORPHOUS WAX FROM PETROLEUM OILS

Application filed July 16, 1927. Serial No. 206,333.

This invention relates to improvements in treating petroleum oils and fractions thereof containing amorphous paraffine wax to effect the removal of the wax therefrom while in the amorphous state. The process of the invention is of special value and application in the treatment of petroleum oils containing lubricating oil constituents and amorphous wax and provides a simple and economical process adapted to remove the amorphous wax continuously without a preliminary treatment that might impair the lubricating properties of the oil.

In refining petroleum oils for the production of high grade lubricants it is customary to distill off the lighter fractions, such as gasoline, kerosene, etc., with steam or at reduced pressure to avoid cracking of the lubricating oil constituents as cracking reduces the viscosity of lubricating oil constituents and greatly decreases their value. Crude oil reduced in this manner contains amorphous wax which tends to congeal at low temperatures and its presence is therefore objectionable in a lubricating oil because of the high pour test it gives to the oil. It is impossible from a practical standpoint to remove the wax while in an amorphous state from oils of this character in the ordinary filter press due to rapid plugging of the filtering medium. In order to convert the amorphous wax to a crystalline form so that it might be satisfactorily removed by filter pressing a high temperature must be employed which is such as to greatly impair the lubricating properties of the oil.

It has been customary in the past to blend oils of this character with a diluent such as naphtha, chill the blend to an extremely low temperature and allow the naphtha-oil solution to stand for a period of several days to permit the wax to settle during which time the entire body of oil must be maintained at a temperature of about −10 to 0° F. This treatment known as cold settling requires large storage capacity and an excessive amount of refrigeration.

Attempts have been made to effect separation of amorphous wax from oils of this character by centrifugal action, but such operations in the past have been more or less unsatisfactory due to the extremely limited capacity of the machines it has been necessary to employ and to the difficulties encountered in removing the separated wax from the zone of centrifugal action. Such operations in the past have either required the use of a carrier liquid to remove the separated wax by flotation or that the bowl be heated to melt the wax in order that it might be removed in a liquid state. The use of a carrier liquid has the disadvantage that the carrier liquid itself must be chilled to the same temperature as the oil so that a greater refrigerating capacity is required and the wax after separation from the oil must be dehydrated. Further, the presence of a carrier liquid in the bowl of a centrifuge of limited capacity materially decreases the amount of oil that can be handled. Heating the bowl while rotating at high speeds involves difficult problems in heat transfer and increases the refrigerating capacity necessary to maintain the bowl charge at the temperature required for effective separation of the oil and wax.

The present invention provides an improved process whereby amorphous wax may be separated from oils containing it by centrifugal force and the separated wax discharged continuously from the zone of centrifugal action without the use of a carrier liquid or the application of heat to any part of the charge within the zone of centrifugal action. According to the present invention the oil to be treated, which has preferably been given a preliminary treatment such as treatment with 66° Baumé sulfuric acid or filtering, to remove asphaltic constituents and other impurities which act as protective colloids tending to prevent coalescence of the minute wax particles to form larger particles, is blended with a diluent material such as a closely cut low end boiling point naphtha distillate and the blend so produced heated to a temperature of approximately 100° F. to insure all of the wax present passing into solution in the diluent. The mixture is then gradually cooled to a temperature of about −10° F. and mildly agitated during the chilling operation. The chilled naphtha-

oil solution is then subjected to centrifugal force to effect the separation of the wax from the naphtha-oil solution and the separated wax mechanically discharged in a continuous manner from the zone of centrifugal action at one point with movement toward the axis of rotation while the wax free naphtha-oil solution is discharged continuously therefrom in a similar manner at another point.

The blending naptha may be subsequently separated from the wax free oil by distillation and recovered for further use as a diluent.

The process of the invention may be further described in connection with the accompanying drawings which illustrate in a somewhat conventional manner one form of apparatus adapted for the practice of the improved process of the invention, but it is intended and will be understood that this further description is for the purpose of exemplification and that the invention is not limited thereto.

In the drawings:

Fig. 1 is a somewhat diagrammatic representation in elevation of an arrangement of apparatus adapted for the practice of the improved process of the invention.

Fig. 2 is an elevation partly in section of one form of centrifuge suitable for use in the arrangement illustrated in Fig. 1.

Fig. 1 of the drawings shows a storage tank 1 connected through pump 2 and line 3 with a heat insulated mixing tank 4 having a heating coil 5 and stirring device 6 arranged therein. The mixing tank 4 is also provided with a valved inlet connection 7 through which a diluent material such as a naptha distillate may be introduced as desired. Valved draw-off connection 8 and pump 9 are arranged to withdraw liquid from the mixing tank 4 and convey it to the insulated cooling tank 10 provided with coil 11 through which a suitable cooling medium may be circulated. Cooling tank 10 is also equipped with a mechanical stirring device 12. Connections 13, 14 and pump 15 are arranged to supply cool oil from the cooling tank 10 to the centrifuge 16. Valved connection 13a may advantageously be provided to withdraw oil from the upper portion of the cooling tank 10. The stand pipe 17 is interposed in connection 14 to assist in maintaining a positive rate of feed to the centrifuge. Connection 18 is provided to convey effluent liquid from the centrifuge to the storage tank 19. The cold clear solution discharged from the centrifuge may if desired be passed in heat exchanging relations with the fresh oil supplied to the cooling tank 10 before the former is finally discharged to the storage tank. Connection 20 is arranged to direct the solid material discharged from the centrifuge to the conveyor 21.

The centrifuge illustrated in Fig. 2 comprises a bowl 22 supported by supporting web 23 and rotatably mounted on the shaft 24. A spider 25 held in place by the collar 26 is arranged to steady the end of the bowl 22 opposite to the supporting web 23. The element 27 is keyed to the shaft 24 and is provided at its outer surface with an extension 28 which is helical in form and extends approximately to the inner surface of the bowl 22. The central portion of the bowl 22 is cylindrical in form tapering toward the shaft at one end. The other end of the bowl is provided with a ring 29 extending inwardly from the periphery of the bowl. The tapered end of the bowl is continued until it approaches nearer to the shaft 24 than the inner edge of the ring 29. The bowl 22 is provided with a series of openings 30 around its periphery at one end beyond the ring 29. The shaft 24 is supported by the bearings 31 and may be driven directly from any suitable source of power, for example, by a silent chain engaging the sprocket 32. The bowl 22 is driven through supporting web 23 by the sleeve 33, for example, by a silent chain engaging the sprocket 34. In the arrangement illustrated sprocket 34 is slightly larger than the sprocket 32 so that while the bowl 22 and the helix 28 rotate in the same direction, the bowl will rotate at a slightly lower speed. While both the bowl 22 and the helix 28 rotate at high speed the differential between them is relatively small so that any difficulties incident to the provision and care of a high speed bearing between the shaft 24 and the sleeve 33 are eliminated. The direction and speed of rotation of the helix 28 relative to that of the bowl 22 is such that the helix will operate to discharge continuously at the tapered end of the bowl any solid material which would otherwise accumulate therein, for example, the bowl may be driven at 1500 to 1800 R. P. M. and the helix at 1550 to 1850 R. P. M. Collecting rings 34' and 35 are provided at opposite ends of the bowl to collect the wax free naptha-oil solution and separated solid material as they are discharged from the bowl. A stuffing box 36 is arranged to prevent leakage of the material which is introduced through the interior of the hollow shaft 24.

In the operation of the apparatus described and illustrated in accordance with the improved process of the invention the quantity of the oil to be treated for the removal of amorphous wax is introduced to the mixing tank 4 from the storage tank 1 and a diluent such as naptha added to the oil. The most advantageous proportions of oil and diluent material will vary somewhat with the character of the oil and diluent material employed. A blend comprising 40% of the oil to be treated and 60% of a closely cut naptha distillate having an end boiling point of approximately 430° F. will produce satisfactory results in most cases. The naptha-oil mixture is then heated to a temperature of

about 100° F. and thoroughly agitated to insure all of the amorphous wax passing into solution in the naptha distillate. The naptha-oil solution is then transferred to the cooling tank 10 where its temperature is gradually reduced to approximately −10° F. by circulating a cooling medium such as cold calcium chloride brine through the cooling coil 11. The naptha-oil solution is preferably agitated mildly at intervals by means of a mechanical stirring device 12 during the cooling operation. The cool oil is then pumped to the stand pipe 17 from which it is supplied to the centrifuge 16 in a continuous manner. The mixture entering the centrifuge through the hollow shaft 24 passes through the hollow element 27 and is discharged to the interior of the rotating bowl 22 through a series of openings 38 where it is subjected to centrifugal force. The centrifugal force to which the mixture is subjected is many times greater than the force of gravity and causes the wax which is heavier than the naptha-oil solution to separate therefrom forming an outer layer which is mechanically conveyed to the tapered end of the bowl by the helix 28 where it is discharged from the bowl continuously. The motion of the helix relative to the bowl is not sufficient to act positively on the liquid, due to its greater fluidity so that the inner layer of clear cold liquid will flow through the bowl axially in a direction opposite to the motion of the outer layer of wax, passing over the inner edge of the ring 29 as it is displaced by fresh oil supplied to the bowl. As the wax is mechanically conveyed by the helix 28 to a point nearer the axis of rotation than the point at which the naptha-oil solution is discharged, while still subjected to the action of centrifugal force, the last traces of the oil or naptha-oil solution may be separated therefrom so that a maximum yield of wax free oil and a high melting point wax may be obtained. The speed at which the bowl may most advantageously be driven will vary somewhat with the character of the oil, the rate at which the oil is introduced thereto and the length of the bowl employed. In general, a lower speed is permissible with a longer bowl and with a decrease in the rate of introduction of fresh oil. With a bowl 15″ in diameter and 45″ long a rotative speed of 1500 R. P. M. will in most cases be sufficient to effect satisfactory separation of the wax from a naptha-oil solution supplied thereto at a rate of 2000 to 3000 gallons per hour, though higher speeds may be employed to insure the separation of the last traces of wax. Where higher speeds are employed a higher melting point wax will be obtained.

Where a refinery has a plant equipped for the old gravity method of cold settling the process of the invention may be employed in conjunction therewith. For example, the cooled solution may be allowed to stand for a time before centrifuging to effect a preliminary concentration of the wax by settling. A layer of cool clear liquid may then be drawn off from the upper part of the settled liquid body and the remainder of the naptha-oil solution containing the wax concentrated therein by settling, centrifugally treated as above described.

In refineries which are not provided with the equipment for present day cold settling methods the various oils or solutions now subjected to the cold settling method of treating may be treated continuously in accordance with the present invention while avoiding heavy investment in cold settling tanks and excessive refrigerating capacity. The process of the invention has the further advantages that the apparatus required is of rugged construction and does not require critical adjustments or the supervision of a skilled operator.

I claim:

1. The improvement in the treatment of petroleum oils for the removal of amorphous wax contained therein, comprising chilling the oil to be treated in the presence of a low boiling diluent and subjecting the chilled mixture to centrifugal force, whereby the wax and solution of wax free oil and diluent are separated into layers, mechanically discharging the wax in a continuous manner from the zone in which it is subjected to centrifugal force at one point with movement toward the axis of rotation, and continuously discharging the wax free oil and diluent material from said zone with movement toward the axis of rotation at another point.

2. The improvement in the treatment of petroleum oils for the removal of amorphous wax contained therein, comprising chilling the oil to be treated in the presence of a low boiling naptha distillate and subjecting the chilled mixture to centrifugal force whereby the wax and wax free naptha-oil solution are separated into layers, mechanically discharging the wax in a continuous manner from the zone in which it is subjected to centrifugal force at one point with movement toward the axis of rotation and continuously discharging the wax free naptha-oil solution from said zone with movement toward the axis of rotation at another point.

3. The improvement in the treatment of petroleum oils for the removal of amorphous wax contained therein, comprising chilling the oil to be treated in the presence of a low boiling naptha distillate, subjecting the chilled mixture to centrifugal force, whereby the wax and wax free naptha-oil solution are separated into layers, causing the wax to move axially in the zone in which it is subjected to centrifugal force and causing the wax free naptha-oil solution to move axially in said zone in the opposite direction, mechanically discharging the wax in a continu-

ous manner from the zone in which it is subjected to centrifugal force at one point with movement toward the axis of rotation and continuously discharging the wax free naptha-oil solution from said zone with movement toward the axis of rotation at another point.

4. The improvement in the treatment of petroleum oils for the removal of amorphous wax contained therein, comprising heating the oil to be treated in the presence of a low boiling naptha distillate and subsequently chilling the admixed oil and diluent to a temperature below 0° F., subjecting the chilled mixture to centrifugal force whereby the wax and wax free naptha-oil solution are separated into layers, mechanically discharging the wax in a continuous manner from the zone in which it is subjected to centrifugal force at one point with movement toward the axis of rotation and continuously discharging the wax free naptha-oil solution from said zone with movement toward the axis of rotation at another point.

5. The improvement in the treatment of petroleum oils for the removal of amorphous wax contained therein, comprising chilling the oil to be treated in the presence of a low boiling naptha distillate, subjecting the chilled mixture to centrifugal force, whereby the wax and wax free naptha oil solution are separated into layers, continuously discharging the wax free naptha-oil solution from the zone in which it is subjected to centrifugal force at one point with movement toward the axis of rotation, and mechanically discharging wax in a continuous manner from said zone with movement toward the axis of rotation at a point nearer to said axis than the point at which the wax free naptha-oil solution is discharged.

6. The improvement in the treatment of petroleum oils for the removal of amorphous wax contained therein, comprising chilling the oil to be treated to below 0° F. in the presence of a low boiling diluent, subjecting a body of the chilled mixture to a settling operation subsequently drawing off a layer of clear liquid from the upper portion of said body, withdrawing a concentrated mixture from the lower portion of said body and subjecting the mixture so withdrawn to centrifugal force whereby the wax and solution of wax free oil and diluent material are separated into layers, mechanically discharging the wax in a continuous manner from the zone in which it is subjected to centrifugal force at one point with movement toward the axis of rotation and continuously discharging the wax free oil and diluent material from said zone with movement toward the axis of rotation at another point.

7. The improvement in the treatment of petroleum oils for the removal of amorphous wax contained therein, comprising chilling the oil to be treated in the presence of a low boiling naptha distillate to a temperature below 0° F., subjecting the chilled mixture to centrifugal force whereby the wax and wax free naptha-oil solution are separated into layers, mechanically discharging the wax in a continuous manner from the zone in which it is subjected to centrifugal force at one point with movement toward the axis of rotation and continuously discharging the wax free naptha-oil solution from said zone with movement toward the axis of rotation at another point.

8. The improvement in the treatment of petroleum oils for the removal of amorphous wax contained therein, comprising chilling the oil to be treated in the presence of a low boiling diluent and subjecting the chilled mixture to centrifugal force in a separating zone, whereby the wax and solution of wax free oil and diluent are separated into layers, causing liquid oil from which wax has been partially separated to move axially in the separating zone while subjected to the action of centrifugal force, mechanically conveying wax subsequently separating from the partially clarified oil axially in the oppositely direction, continuously discharging the wax from the zone in which it is subjected to centrifugal force at one point with movement toward the axis of rotation, and continuously discharging the wax free oil and diluent material from said zone with movement toward the axis of rotation to another point.

In testimony whereof I affix my signature.

PIERCE MASON TRAVIS.