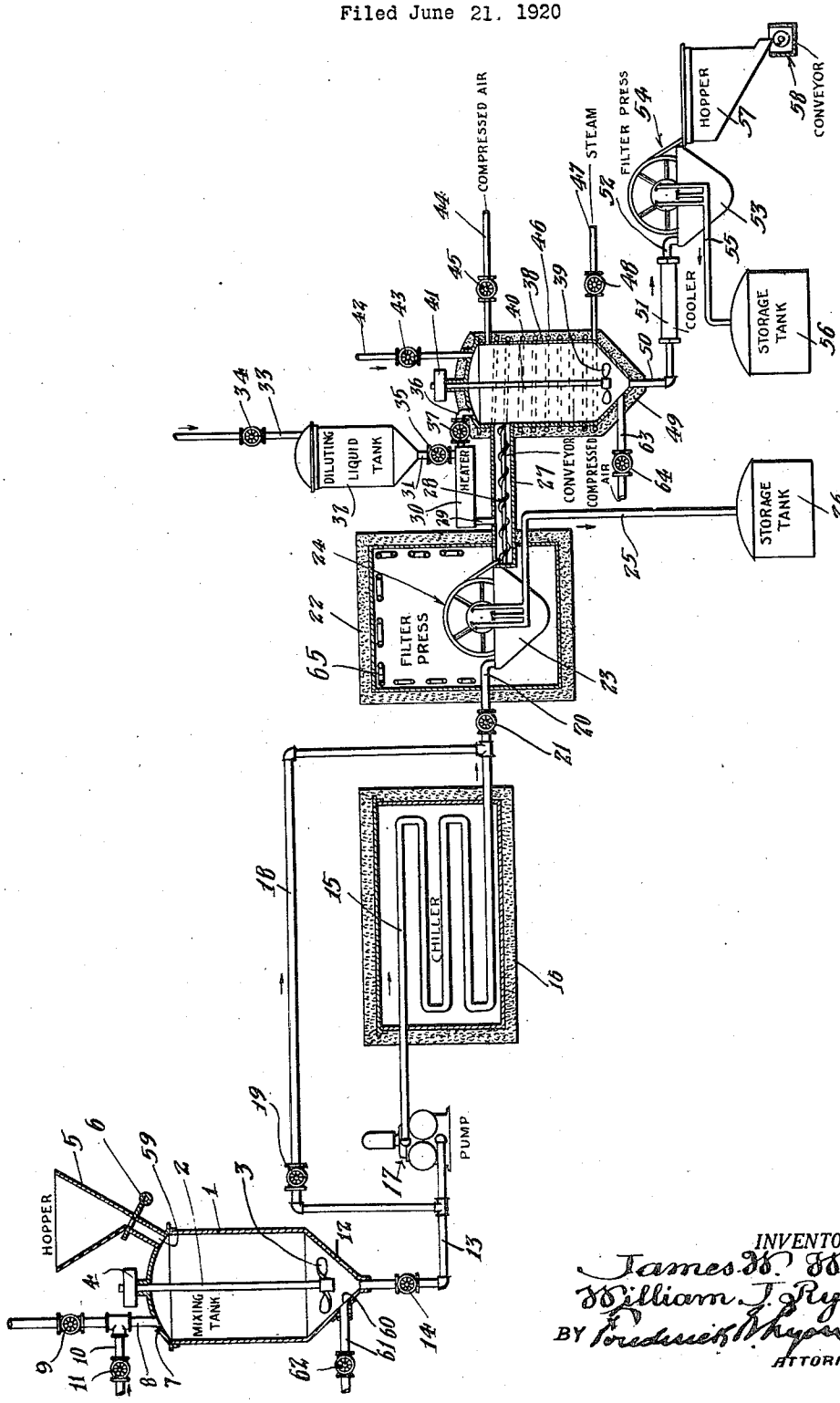
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PROCESS FOR LOWERING THE COLD TEST OF LUBRICATING OILS

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PROCESS FOR LOWERING THE COLD TEST OF LUBRICATING OILS.

Application filed June 21, 1920. Serial No. 390,417.

To all whom it may concern:

Be it known that we, JAMES W. WEIR and WILLIAM J. RYAN, Jr., citizens of the United States, both residing at Fillmore, in the county of Ventura and State of California, have invented a new and useful Process for Lowering the Cold Test of Lubricating Oils, of which the following is a specification.

This invention relates to a process for effecting the separation of paraffin wax, amorphous wax, gummy or gelatinous substances, or hydrocarbons which normally solidify or become semi-solid below a temperature of approximately 140° F. from mineral, vegetable or animal oils, or hydrocarbons normally liquid at the same temperature.

An object of the invention is to lower the cold test of lubricating oils so that they will not solidify at comparatively low temperatures.

Another object of the invention is to effect the separation expeditiously and inexpensively.

Another object is to provide a continuous process or one that is semi continuous by the admixture of a wax-adherent carrying medium with the oil to be treated, or with a diluted solution containing the oil, at a reduced temperature and then passing the wax-adherent medium, after the wax or other solid or semi solid hydrocarbons have adhered to said medium, through a filter of any suitable type capable of extracting or separating the wax-adherent medium and its adhering hydrocarbons from those hydrocarbons which are liquids at the temperatures and pressures under which the liquid hydrocarbons pass through the filter.

The accompanying drawing is a more or less diagrammatic view, largely in section, of an apparatus capable of performing the various operations incident to the process.

The apparatus illustrated in the drawing is only typical and is shown and will be described merely for the purpose of aiding in understanding the process, and it is to be understood that any other suitable apparatus may be substituted for that shown and described to effect the process. The apparatus shown in the drawing is constructed as follows: There is provided a mixing tank 1 having in its interior a shaft 2 provided with mixing paddles 3. The shaft projects through the upper end of the tank 1 and is provided on its projecting end with a pulley 4 adapted to be operated by being belted to any suitable prime mover. The paddles 3 serve to properly stir, beat and mix the substances discharged into the tank. The upper end of the tank is provided with an inlet 59 with which communicates a receiving hopper 5 provided with a valve 6. The upper end of the tank 1 is provided with another inlet 7 with which communicates tubing 8 having a valve 9. Connected with the tube 8 is a branch tube 10 having a valve 11. The tank 1 is provided at its lower end with a third inlet 60 with which connects a tube 61 provided with a valve 62. The lower end of the tank 1 forms a cone 12 from which discharges a pipe 13 provided with a valve 14.

The pipe 13 may be connected directly with the coil 15 of a chiller, indicated in general by the character 16 and constructed in any suitable manner, and the contents of the tank 1 may be discharged by gravity into the coil 15. However, in the drawing, I have connected the pipe 13 with a suitable pump 17 and the pump is connected with the coil 15, so that, if desired, the contents of the tank 1 may be pumped through the coil 15.

The discharge end of the coil 15 is connected by a tube 18 with the pipe 13 and the tube 18 is provided with a valve 19. The coil 15 connects with a nozzle 20 provided with a valve 21. The nozzle 20 passes through the wall of an insulated cold chamber 22 and discharges into the tank 23 of a filter press indicated in general by the character 24. This filter press may be of any known or preferred type employing vacuum or pressure. The filtrate discharge pipe from the filter press 24 is indicated at 25 and is connected with a storage tank 26.

The solids or semi-solids discharge from the filter press 24 into the trough 27 of a conveyor, the conveyor screw being indicated at 28. With the trough 27 is connected the discharge nozzle 29 of a heater 30 which is connected by a pipe 31 with a tank 32 adapted to hold a suitable diluting or washing liquid supplied to the tank through a tube 33 provided with a valve 34. The pipe 31 is provided with a valve 35. The heater 30 is provided with a second discharge nozzle 36 having a valve 37.

The conveyor trough 27 and the nozzle 36 both discharge into a suitable insulated washing tank 38 provided in its interior with stirring paddles 39 on a shaft 40 which projects through the upper end of the wash-

ing tank and which is provided with a pulley 41 whereby the shaft 40 may be connected by a belt to any suitable prime mover for operating the paddles 39. Connected with the washing tank 38 is a steam supply pipe 42 having a valve 43. Also connected with said tank are compressed air supply pipes 44, 63 having valves 45, 64, respectively. The washing tank 38 is provided with a heating coil 46 adapted to be supplied with steam by a pipe 47 having a valve 48. The lower end of the washing tank 38 forms a cone 49 from which discharges a pipe 50 connected with a suitable cooler 51 that is provided with a discharge nozzle 52 arranged to discharge into the tank 53 of a filter press, indicated in general by the character 54. The filter tank 54 may be of any suitable type and the filtrate discharges from the filter press through a pipe 55 which empties into a storage tank 56.

The filter press 54 is arranged to discharge the solids and semi-solids therefrom into a suitable bin or hopper 57. The hopper 57 is arranged to discharge its contents into a suitable conveyor 58 which, in this instance, is of the screw type.

In practice, the process is performed with the hereinbefore described apparatus as follows:

The hydrocarbons to be treated for removal of the paraffin wax, amorphous wax, gummy or gelatinous substances or, in other words, those hydrocarbons which normally solidify or become semi-solid below a temperature of approximately 140° F., are admitted into a suitable mixing tank such as, for example, the tank 1 through the tube 8, the valve 9 being opened for this purpose. The mixing tank 1 will also be supplied with a diluent such, for example, as naphtha, alcohol or any other liquid suitable for lowering the cold test and viscosity and decreasing the specific gravity of the hydrocarbons being treated. The diluent is mitted to the tank 1 through the tubes 10, 8 by opening the valve 11.

Then a wax-adherent carrying medium will be supplied to the tank 1 through the hopper 5, the valve 6 being opened to admit the medium to the tank. The carrying medium may be of any suitable character capable of permitting or inducing the adhesion of wax thereto and examples of such mediums are diatomaceous earth, fuller's earth, clay, sand, saw-dust or any other comminuted solid or porous substance or any fibrous mineral, vegetable or animal substance. Diatomaceous earth, in a finely powdered condition such as that found, for example, in California deposits, is particularly well adapted for the purpose.

The naphtha or other diluent is added to the hydrocarbons in a liquid condition at the temperatures employed in the process. The diluent, hydrocarbons and wax-adherent medium are thoroughly mixed in the tank 1 and the mixing may be effected in any suitable manner. One way of effecting the mixing is by operation of the paddles 3, or air is admitted to the tank through the tube 61 by opening the valve 62, or the paddles and air can both be used. The paddles and air function to agitate the various ingredients of the mixture. The ingredients are kept from settling by constantly stirring or agitating the mixture.

After mixing of the diluent, hydrocarbons and wax adherent medium is effected, the valve 14 is opened, thus allowing the mixture to flow through the pipe 13 into the chilling coil 15. This may be effected by gravity or the pump 17 may be operated to pump the mixture through the chilling coil. The mixture will be chilled in the coil 15 sufficiently to cause the amorphous wax or other hydrocarbons which are to be removed to congeal and cling to the surfaces of the particles of diatomaceous earth or other wax-adherent medium employed.

The valve 21 will be opened thus allowing the chilled mixture to discharge into the filter 24. The filter 24 illustrated is of the type known as a filter press and it may be of the pressure type or the vacuum type. If the vacuum type of filter press is employed the process will be continuous, and if the pressure type of filter press is employed the process will be partially continuous. The mixture in the filter press 24 is maintained at the desired temperature because of the action of the cooling coils 65 in the cold chamber 22.

In the filter press 24 the diluent and the hydrocarbons, which are liquid at the temperature employed, are separated from the solids and semi-solids, said diluent and liquid hydrocarbons flowing through the pipe 25 into the tank 26, the wax-adherent medium together with the wax adhering thereto being deposited upon the filtering surfaces of the filter press and forming a cake which discharges into the conveyor trough 27. The oil received in the tank 26 is separated from the diluent also received by said tank by the well known methods of distillation and, if the oil is to be used as a lubricant, it may be reduced to any desired consistency.

The tank 32 will be supplied with a suitable solvent or washing liquid through the pipe 33 by opening the valve 34. The solvent may be naphtha, alcohol or any other liquid capable of dissolving the wax or other congealed hydrocarbons forming the cake. The valve 35 will be opened to allow the solvent to pass into the heater 30 which thus functions to heat the solvent. The heated solvent discharges through the tube 29 into the trough 27 and is mixed with the cake in said trough, the mixing being accom-

plished by the conveyor screw 28; or the valve 37 will be opened so that the heated solvent will be discharged through the tube 36 directly into the washing tank 38; or the heated solvent may be discharged into both the trough and said tank.

The cake, together with whatever solvent has been admitted to the conveyor trough 27 and mixed therewith, will be transmitted by the conveyor screw 28 to the washing tank 38 where the washing operation is performed. To assist in the washing operation the paddles 39 may be operated or air may be admitted through the tube 63 by opening the valve 64 or the paddles and air may both be employed. The cake is dissolved by the heated solvent and the dissolving action is assisted by the stirring, mixing or agitation of the cake and solvent in the washing tank 38.

The mixture thus formed in the tank 38 includes the amorphous wax or other hydrocarbons which were separated by the filter 24 from the oil and diluent received by the tank 26, and said mixture also includes the wax-adherent medium and the solvent, and the next operation is to separate the wax and solvent from the wax-adherent medium and this operation will now be described.

The mixture flows from the tank 38 through the pipe 50 to the cooler 51 where the mixture is reduced to the desired temperature, such temperature preferably being sufficiently low to prevent vaporization of the hydrocarbons. From the cooler 51 the cooled mixture discharges through the nozzle 52 into the filter 54 which, in this instance, is of the type known as a filter press of either the pressure or vacuum type. This filter press 54 separates the wax-adherent medium from the liquid, the liquid being a mixture of the solvent and the amorphous wax or other hydrocarbons which congeal or solidify at a temperature less than approximately 140° F. The liquids flow from the filter through the pipe 55 into the tank 56, and the washing liquid or solvent is separated from the wax or other extracted hydrocarbons by any well known methods of distillation and refining. The wax-adherent medium forms a cake in the filter press 54 and such cake is discharged into the hopper 57 and from said hopper the wax-adherent medium discharges into the conveyor 58 which conveys the wax-adherent medium to any desired place of deposit. This wax-adherent medium may then be reemployed as before for effecting the deposition of wax upon the surfaces thereof, which is to say, that the wax-adherent medium may be again introduced to the tank 1 through the hopper 5 to make another cycle. The wax-adherent medium may thus be used over and over.

If it be desired to temporarily discontinue operation of that portion of the apparatus to the right of the valve 21, said valve will be closed and the valve 19 will be opened to permit circulation of the cooled mixture through the chilling coil 15, pipes 18 and 13, so that the mixture will not clog this portion of the apparatus, as would result if such circulation were not provided for when the valve 21 is closed.

From the foregoing it will be understood that the essential operations of the process are the mixing together of a wax-adherent medium and the hydrocarbons containing the wax which is to be removed, chilling the mixture to congeal the wax, and then filtering the chilled mixture to separate the uncongealed hydrocarbons from the wax and wax-adherent medium. The other operations described are important for working the process on a commercial basis and have therefore been described in detail.

It is to be understood that the term "wax" employed in this specification generically defines any of the hydrocarbons which congeal or solidify or cease to be liquid at a temperature not substantially higher than 140° F.

In practice the above described invention has been employed under the following conditions and with the results here stated:

By well known methods of distillation, under a sufficient reduced pressure to prevent a substantial cracking, crude petroleum oil produced from wells in Ventura County, California, having a gravity of 31° Baumé and a paraffin wax content of 6.4%, was used to obtain a lubricating oil distillate fraction, which was 32% by volume of the crude oil distilled. This lubricating oil distillate commonly called "wax distillate," containing 20% of paraffin wax, was reduced by the well known method of distillation with fire and steam to a 31½% of bottom, which was equivalent to 10% of the crude petroleum oil from which it was obtained.

This reduced lubricating oil stock was treated at the rate of 25 pounds of 66° Baumé sulfuric acid per barrel of oil, by well known methods, giving a finished yield of 75% of the stock so treated, which was equivalent to 7½% of the crude petroleum oil from which it was obtained. The viscosity of this acid treated lubricating oil stock was 58 seconds at a temperature of 210° Fahr., Saybolt viscosimeter; the paraffin wax content 25%, substantially all of said paraffin wax content being in the amorphous form.

To this acid treated lubricating oil stock was added a petroleum oil diluent which had a gravity of 55° Baumé, making a mix of 25 parts by volume of acid treated lubricating oil stock to 75 parts by volume of diluent. The viscosity of this mix was 31

seconds at 100° Fahr., Saybolt viscosimeter.

To this diluted acid treated lubricating oil stock was added one half pound of a wax adherent medium, diatomaceous earth, per gallon of diluted oil stock. This mixture of lubricating oil stock, diluent, and wax adherent medium was then thoroughly commingled by means of mechanical agitation and then passed under pump pressure through a Gray chiller of well known type, where the temperature was reduced to 0° Fahr. At this reduced temperature the paraffin wax content of the mixture changed from the liquid to the solid and semi-solid state, and while at this reduced temperature, 0° Fahr., and under a pump pressure of approximately 75 pounds gauge pressure this chilled mixture was passed into a Kelly filter press of the well known type commonly used for separating solids from liquids, where the lubricating oil stock and diluent was separated from the congealed paraffin wax content, which remained adhering to and disseminated throughout the wax adherent medium forming a filter cake on the plates of the press; the lubricating oil stock and diluent remaining liquid passed through the filter cake and filter cloth into a pipe which leads to the receiving tank. The filter cake containing the paraffin wax (6¼% of the diluted lubricating oil stock) was conveyed by means of a conveyer into a receiving tank and mixed with a sufficient amount of a hot petroleum oil distillate which dissolved the paraffin wax-content contained therein, and the dissolved paraffin wax was separated from the said petroleum oil distillate solvent and wax adherent medium by a subsequent filtration and distillation well known in the art.

The diluent and lubricating oil stock separated from the paraffin wax and wax adherent medium was then subjected to a distillation process and the lubricating oil separated from the diluent. The lubricating oil thus separated from its paraffin wax content was 5.6% of the original crude petroleum oil from which it was made, and has a viscosity of 60 seconds at 210° Fahr. Saybolt viscosimeter. The per cent of paraffin wax separated from the lubricating stock was 1.9% of the crude petroleum oil. This example of an actual operation carried on in employment of this invention is for illustrative purposes only and the invention is not limited thereto.

We claim:

1. The process of lowering the cold test of lubricating oils comprising a mixing diluted lubricating oil stock with comminuted wax adherent material, passing the mixture through a chiller to chill the mixture sufficiently to congeal its wax content, the diluted stock consisting of lubricating oil stock together with lower boiling point hydrocarbons and producing with the wax adherent material a mixture pumpable at the wax congealing temperature, filtering the chilled mixture to separate the diluted stock from the wax adherent medium and congealed wax, and separating the lower boiling point hydrocarbons from the lubricating oil stock.

2. The process of lowering the cold test of lubricating oils which consists in combining with lubricating oil stock containing wax, a diluent and additionally adding thereto a comminuted wax adherent medium, then chilling the mixture thus produced to congeal the wax upon the wax adherent medium, the diluted stock other than the wax remaining liquid and then separating the chilled associated wax and wax adherent medium from the liquid mixture.

3. The process of lowering the cold test of lubricating oils by separating hydrocarbons which normally solidify or become semi-solid below a temperature of approximately 140° F., from hydrocarbons which remain liquid at that temperature which consists in adding a diluent and comminuted hydrocarbon adherent medium to lubricating oil stock, then chilling the mixture sufficiently to congeal the hydrocarbons which solidify or become semi-solid below the temperature of approximately 140° F., the other hydrocarbons in the mixture remaining liquid, then filtering the liquid hydrocarbons from the solidified hydrocarbons and hydrocarbon adherent medium and maintaining the mixture at the chilling temperature during the filtering operation.

4. A process of lowering the cold test of lubricating oils which consists in combining with lubricating oil stock containing wax, a wax adherent medium and a diluent, chilling the mixture thus produced to congeal the wax upon the wax adherent medium, the diluted stock other than the wax remaining liquid and then separating the chilled associated wax and wax adherent medium from the liquid mass.

5. A process of lowering the cold test of lubricating oils which includes the operations of combining with lubricating oil stock a diluent and a wax adherent medium, chilling the mixture thus produced to congeal the wax upon the wax adherent medium, the diluted stock other than the wax remaining liquid, then separating the chilled associated wax and wax adherent medium from the liquid mass, then dissolving the wax that has been congealed on the wax adherent medium with a solvent and separating the wax solution from the wax adherent medium.

6. A process of lowering the cold test of lubricating oils which includes the operation of combining with lubricating oil stock, while the wax is dissolved therein, a com-

minuted wax adherent medium, chilling the mixture thus produced to congeal the wax upon the wax adherent medium, the diluted stock other than the wax remaining liquid, separating the chilled associated wax and wax adherent medium from the liquid mass, then heating and dissolving with a suitable solvent the chilled associated wax, and finally separating the wax adherent medium from this solution whereby the wax adherent medium may be re-used in a similar cycle of operations.

Signed at Los Angeles, California this 15th day of June 1920.

JAMES W. WEIR.
WILLIAM J. RYAN, JR.

Witnesses:
GEORGE H. HILES,
L. BELLE WEAVER.