

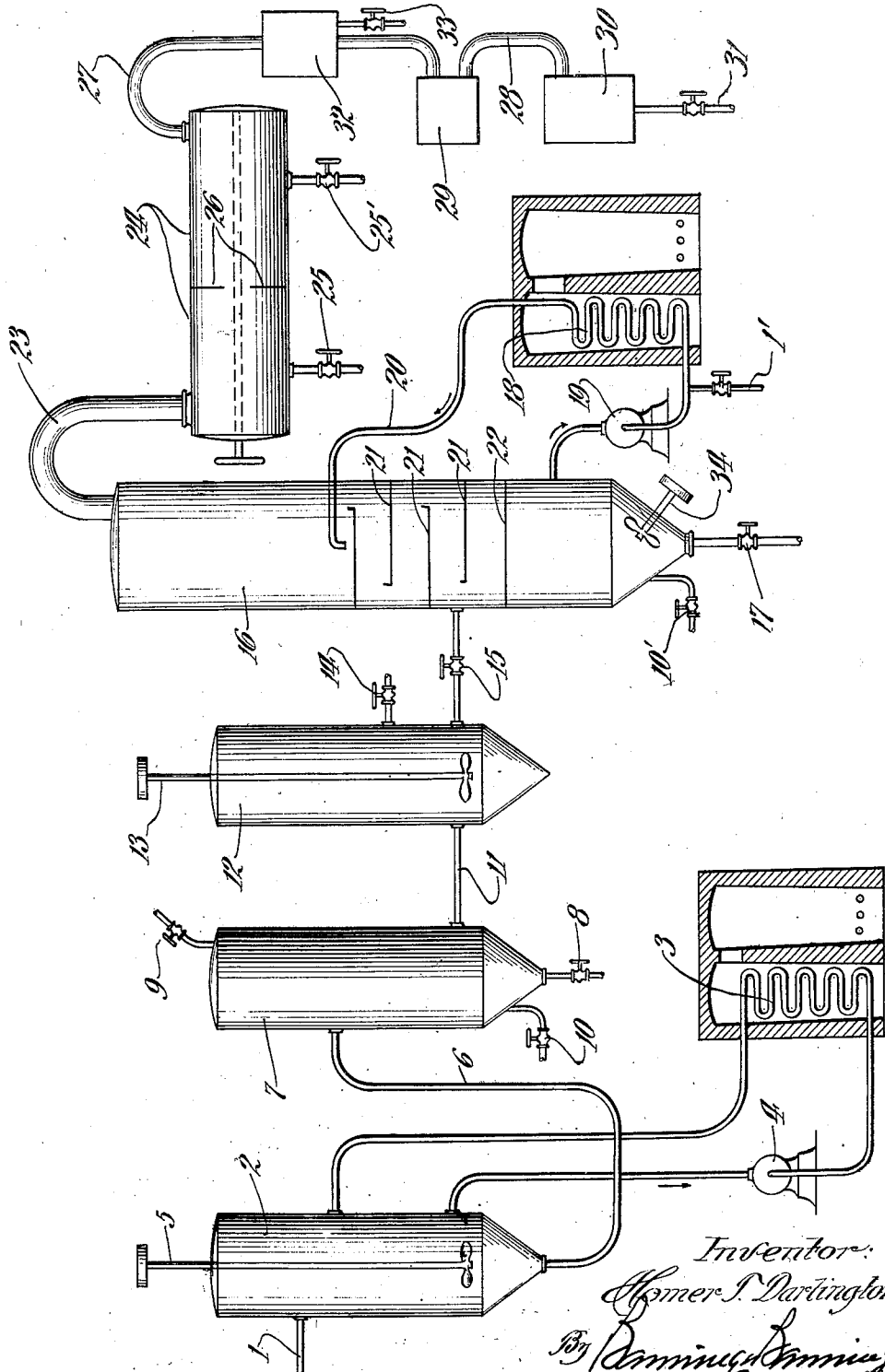
Oct. 25, 1932.

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1,884,587

PROCESS FOR THE PRODUCTION OF LUBRICATING OIL

Filed May 26, 1927



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UNITED STATES PATENT OFFICE

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PROCESS FOR THE PRODUCTION OF LUBRICATING OIL

Application filed May 26, 1927. Serial No. 194,292.

5 An object of my invention is to take out of the oil during the process of manufacture carbon more readily separable to the end that there will be less carbon to deposit, and, therefore, less deposited while the oil is being used as a lubricant.

10 An object of my invention is to produce a lubricating oil less liable to deterioration under conditions of use than any now available. Another object of my invention is to remove from the lubricating oil during its manufacture, or stabilize in it, component parts of it of minor value as lubricants, which tend to cause deterioration.

15 Another object of my invention is to produce a lubricating oil of low volatility and superior pouring quality; that is, having a low pour test. Another object of my invention is to provide a means for producing standardized lubricating oils of definite, predetermined quality and character as a direct result of distillation and allied operations without subsequent manipulation.

20 Another object of my invention is to produce lubricating oil of the character and in the manner stated from deteriorated oil, such as oil drained from automobile crank cases, and those qualities and kinds of petroleum that are usually deemed unfit for the purpose.

25 Another object of my invention is to provide an apparatus that can be readily and effectively used in the practice of my said processes.

30 It is well known that lubricating oil when used where it is subjected to heat approaching its volatilization point is darkened by the formation of carbon within the oil. This is particularly true of oils used in bearings, and is frequently observed with respect to automobile crank case oil. It is also a matter of common knowledge that lubricating oils when volatilizing leave substantial deposits of carbon on the walls of automobile and other explosive engines.

35 Lubricating oil is used in machinery lubrication where it is in continuous contact with moving metal loses some of its lubricating power. This decrease in lubricating value, is, I believe, the result of changes in the oil

due to contact with the metal and to frictional heat evolved not only by the movement of the metal parts, but also and perhaps primarily by the movement of the molecules within the oil itself. My observation and experience convinces me that this deterioration in lubricating oil is to a large extent due to cracking during such use; that the change is confined to certain relatively unstable fractions of the oil, the major part of the lubricant remaining unchanged.

40 In the distillation of lubricating oil stocks, as usually done, these minor constituents which materially limit the useful life of the lubricant distill and condense with the more useful and desirable constituents. I have found that I can treat lubricating oil stock prior to or during distillation with certain colloidal or quasi-colloidal materials, and by holding them dispersed through the oil during distillation or other treatment remove or hold back and prevent the volatilization in large part of constituents undesirable in lubricating oil, and particularly of free or easily separated carbon.

45 In carrying out the art of my invention, I take lubricating oil that has been used, or crude oil, or heavier fractions thereof, or residue, or mixtures of some or all of them, and intimately commingle the oil stock thus provided with colloidal or quasi-colloidal material, such as bentonite, bentonite and water, hydrated lime, sponge iron, siliceous residue of acid treated clay or serpentine, or mixtures of salt water and clay, or colloid material having metallic coatings, all of which materials or mixtures thereof are hereinafter, for convenience sake, referred to as adsorptive addition agents.

50 I maintain the colloid addition agent in suspension and incorporation in the oil and desirably colloiddally dispersed by mechanically induced circulation and agitation, while I heat it to a temperature above that at which I subsequently distill it. The temperature I so use may vary and may be any temperature between 500° and 850° F., both inclusive, and I may carry out the treatment at normal or higher pressure, either in the same vessel or apparatus in which I sub-

sequently distill, or in a separate vessel or apparatus, subsequently transferring to the distilling unit.

I may at any desired point in the process remove by settling or filtration any of the colloid addition agent, and may repeat the treatment with other colloidal addition agent. I may also at any time remove carbon that has settled out, and I may also before the volatilization of the lubricating oil remove such lighter products as are separated by the preliminary treatment.

By this pre-treatment, which is preferably performed under super atmospheric pressure and at a temperature which will vaporize the more volatile constituents without vaporizing the major portion of the oil stock, I not only remove from the oil stock the free carbon and that which is more readily dissociated, but I vaporize and, as a rule, remove some components of my materials which are more volatile, and I stabilize others by combinations effected when carbon is disassociated, as already stated. Thus I render the lubricating oil subsequently formed from material so treated less likely to form and deposit carbon. I also decrease the volatility of such lubricating oil and minimize it to an appreciable degree to the deteriorating effects of the conditions under which it is used.

One deteriorating influence of considerable importance in the use of lubricants is the dust and dirt, and the fine particles of metal ground from bearings that find lodgement in the oil. These, as has already been intimated, attack certain components of the oil and cause deteriorating changes in them. By dispersing colloid addition agents in the oil stock during pre-treatment, I subject it to conditions akin in effect, though they may differ in characteristics, to those prevailing when the lubricating oil is in actual use, and removing or transforming those components that readily deteriorate, I thus reduce to a minimum the possibility of the oil deteriorating in actual use. The colloidal addition agent in connection with the heat treatment may be employed to coagulate colloidal carbon usually found in still residues and heavy oils. The change of state causes the oil to evaporate at lower temperatures and results in larger proportionate production.

There are usually in petroleum hydrocarbon compounds which when they remain, as they usually do, in lubricating oil, form gums and resins which when the oil is used as a lubricant are deposited on bearings and other parts and are very objectionable. I have found that air or oxygen charged into heated oil stock containing a dispersed colloid addition agent acts upon the gum—and resin—forming compounds in such manner, apparently by oxidization, that thereafter objectionable gums and resins are not formed or deposited, and I have found that the

charged air or oxygen works this change satisfactorily and adequately only when the oil is heated and when colloid agents are also present.

Having removed, held back or transformed the undesirable constituents in my lubricating stock in the manner set forth, I proceed to evaporate and fractionally, selectively condense such portions of the lubricating stock as I may desire. I may or may not add fresh colloid addition agent to the oil before or during distillation, and generally I prefer to do so. I also preferably, though not invariably, use heat for vaporizing lower than that used in the first treatment, so that any material held by the colloidal addition agent will not break up chemically and their products be found in the fractions condensed.

To accomplish distillation at a low temperature, I heat, distill and condense in a vacuum. I am aware that distilling and otherwise treating oil in vacuum is old and well known, and has been practiced in the art for many years. I prefer to use a vacuum such as has been used in actual practice for many years as distinguished from a theoretical vacuum or one that might be practical in laboratory work, but not under actual productive working conditions. I do not, however, restrict myself to a vacuum of any particular degree.

Other features of my process will be made clear in connection with the description which follows of an apparatus which I have invented for used in practicing this process. While this apparatus is peculiarly adapted to the process, its use is not confined to the process, and the process may be performed with any apparatus adapted or adaptable to its various operations.

In the drawing:

A suitable combination of apparatus is shown somewhat diagrammatically.

Reference Figure 2 shows a chamber in which oil stock, together with one or more colloid addition agents, is agitated and mixed by paddle 5 and thoroughly commingled. The oil stock and the colloid addition agent or agents are introduced to the chamber 2 through the pipe line 1, or the like, by a pump (not shown) or by other suitable means, and is circulated for heating by a pump 4 through the coils 3 heated in a furnace as shown, or by any other suitable means. 7 is a settling chamber with which the chamber 2 is in communication by a pipe line 6. It is provided with means for drawing off carbon 8 and with a valve controlled outlet for vapors 9.

12 is a second mixing chamber, which I call a colloid mixer, connected by the transfer line 11 with the settling chamber 7. It has a stirrer of any suitable type, shown as a paddle 13, and also has valve controlled means 14 for the optional introduction of additional colloid agent. The settling chamber also has

means 10 for introducing air for treating gum and resin forming compounds.

16 is a vaporizer in communication by the valve controlled pipe line 15 with the mixing chamber 12. It contains distribution plates 21 to provide extensive vaporizing surfaces, and has in connection with it a pump 19, or other suitable means of circulating liquids from it at a point below the variable liquid level therein 22 through the coil 18 heated by a furnace as shown, or other suitable means, and returning the liquid to the vaporizing chamber 16 at a point above said distribution plates 21.

A pipe or similar means 1' for the introduction of oil stock and colloid addition agent are provided intermediate the pump 19 and the heating means 18. Valve controlled means 17 are provided for removing carbon and sedimentary deposits, and a pipe or the like 10' is provided for the introduction of air for treating gum and resin forming compounds when oil stock that has not been preheated is used, and an agitator 34 for use when addition agent is introduced to the vaporizer 16 through the means 1'.

The vaporizing chamber 16 is connected by suitable means 23 to a controlled selective dephlegmator 24. This dephlegmator 24 contains selective separator plates 26 of such number as may be desired, and pipes or the like 25 and 25' for the selective withdrawal of condensate, two or more in number, according to the number of cuts in finished products desired. The withdrawal pipes 25 and 25' are each controlled either by a valve, or, if preferred, thermostatically.

Connected with the dephlegmator 24 by connections 27 and 28 are a mercury vapor pump, or an inspirator 29, and any other suitable means of creating a vacuum 30, with a valve controlled pipe 31 for discharging evacuated gas, and optionally additional means for cooling 32 with an outlet for water 33 may be provided at any desired point. In practical operation and preferably under super atmospheric conditions, as before stated, I introduce oil stock which it is desired to treat intermixed with one or more colloid addition agents (as already defined) through 1 into chamber 2 and immediately pass it through the pump 4 and the heating coil 3 and, after raising it to the desired temperature, back to the chamber 2 where it is thoroughly stirred and commingled. Ordinarily the mixture is circulated for heating only once, but the operation may be repeated one or more times if deemed desirable or necessary.

From the chamber 2 the mixture passes through the pipe line 6 to the settler 7. It is left there for a sufficient time to permit sedimentary matter to precipitate, and I may draw off heavier sedimentary deposits through the pipe 8, provided for that purpose. I may and, if the oil stock used contains ap-

preciable amounts of gum and resin forming compounds, I do admit air through the pipe 10 to the settling chamber 7 to treat such compounds. The heating of the oil stock in the coil 3 is made sufficient to drive out from the body of the oil lighter constituents and to crack more easily decomposed fractions, and as our purpose is to remove such of these as are not by treatment in the chambers 2 and 7 rendered stable, we may and ordinarily do remove them from the settling chamber 7 through the pipe 9.

The remaining oil stock with the dispersed colloid addition agent I pass through the pipe line 11 to the mixer 12. If, as happens when some oil stocks are used, the quantity of carbon deposited or coagulated with the colloid addition agent has been such that enough active carbon free colloid addition agent is not left in the oil stock to effectively treat it, I may introduce more colloid addition agent to the oil in the mixing chamber 12 through suitable pipe or other means 14, and I thoroughly disperse the agent so added and any already in the oil by the agitating means 13 provided for the purpose. From the chamber 12 I pass the oil stock with dispersed colloid addition agent through the pipe line 15 to the vaporizer 16.

The treatment up to this point I designate pretreatment. For it I may substitute operations accomplishing similar results or other operations for preparing the oil stock in any manner deemed desirable, or I may under some conditions and using some kinds of oil stock charge the oil stock into the vaporizer without any pretreatment other than the dispersion in it of colloid addition agent.

I may cut out, either by the valve in line 15, or by dispensing with it entirely, all or any part of the apparatus 1 to 15, both inclusive, anterior to the vaporizer 16, and do such dispersion and any other pretreatment by any means and with any apparatus that may be available, or that under conditions then prevailing may be deemed suitable. In that case, I introduce oil stock with colloid addition agent through the pipe line 1' at a point intermediate the pump 19 and heating coil 18 and under vacuum agitate the oil stock with added addition agent by a paddle 34 or other suitable means.

In every case I circulate the oil containing the colloid addition agent from the vaporizer 16 or the point of admission 1' through the heating coils 18 and thence into the vaporizer in such manner that the oil will spread itself over the distribution plates 21 as it falls into the main body of oil, which has a variable level 22. Moisture and entrained oil which separate out of vapors as they pass upward in the vaporizing chamber 16 also drop back onto the distribution plates 21 and into the main body of oil. The vapors from the vaporizer 16 pass through trans-

mission means 23 to the dephlegmator 24 for cooling and separation.

I prefer to use a dephlegmator placed horizontally or nearly so and equipped with means for regulating and controlling the rate and degree of cooling and for dephlegmating and separating products selectively. I have invented a dephlegmator peculiarly effective and easily adapted for so selectively
10 dephlegmating and separating products for which an application for Letters Patent of the United States, Serial No. 186,459 is now pending. While I prefer to use this dephlegmator, any means of cooling and separating
15 that will admit of separating lubricating products of varying distinctive characteristics independently and selectively may be employed.

I will, however, now outline some of the features of any dephlegmator or kindred apparatus which I deem desirable to accomplish the desired purpose and which differentiate it from those heretofore used. In the first place, the dephlegmator should have as many
20 communicating zones or sections progressively arranged as there are distinctive products or "cuts", so-called, to be made, and each such section or zone should have means for withdrawing the product thereof. In the drawing, I have, for purposes of illustration, divided the dephlegmator 24 into two sections or zones by division plates 26 and have provided an outlet 25 and 25' for each zone or section.

Means should also be provided for progressive positive accelerated cooling to the end that there may be essential differences between the lubricating oil produced in the several sections or zones. The mechanical
40 means of doing this I do not here describe for the reason that they are features of the invention for which I have applied for Letters Patent Serial No. 186,459, as aforesaid.

To produce lubricating oil having properties differing from that produced in the manner set forth, I may cause all or a definite portion of the oil in one or more zones or sections to be passed to and mingled with all
50 or a determined part of the oil in another zone or section, doing this mixing and blending before such oil is withdrawn from the dephlegmator and providing for its withdrawal when properly blended.

I have found in practice that when products of the distillation of petroleum are progressively dephlegmated in communicating zones, products differing in viscosity, flash
60 point and gravity tend to range themselves in different zones each by itself, and that with like materials treated in the same way the same product within a range of allowable variation will invariably be in the same zone.
65 I have also found that the temperature with-

in the zones provides a convenient means for identifying the product.

While I may regulate and control the withdrawal of products from the several zones of the dephlegmator by hand operated valves
70 or other like means, I prefer to operate, regulate and control the withdrawal of products from the dephlegmator by suitable thermostatic means, using any one of the thermostatic devices known and used in the arts that
75 may be adapted to the purpose. Having determined the heat ranges of any desired series of products I can thus automatically separate and withdraw them.

The descriptive outline I have given above
80 of the particular means of dephlegmating and separating is not intended to limit in any manner my invention either of process or apparatus, but merely to indicate effective means of operation.

I have found that certain constituents of lubricating oil as ordinarily made, other than paraffin, which are easily cracked raise the pour test of the oil. These constituents, which are detrimental rather than aids to lubrication, I remove during pre-treatment performed as described, and in this manner I produce a lubricating oil with a lower pour test.

I may use my colloid addition agent in a dry form, but I prefer to wet it with water. I find that water thus used is itself substantially in effect a colloid addition agent. Water when added to hot oil by itself in any appreciable quantity cools the oil and tends
100 to halt and temporarily hinder reactions, but water distributed over the surface of a solid finely divided addition agent when added to hot oil is practically instantly evaporated and transformed to nascent steam and materially aids the reactions taking place in the oil. Just how it does this is not entirely clear, but it seems demonstrable that there is some disassociation of water molecules that provides hydrogen for the production of desirable fractions and some oxidation effects that are beneficial. Water so added, that is, on the surface of a solid colloid addition agent, I find to be very different in its action from introduced steam and much more efficient in producing desirable results.

I claim:

1. The process of producing lubricating oil which comprises treating oil stock in the presence of one or more colloiddally dispersed
120 solid adsorbent addition agents having the catalytic properties of colloidal bentonite under super-atmospheric pressure, by heating to a temperature between 500° and 850°
125 F., both inclusive, circulating and agitating under such pressure and while so heated and at the same time forcing air or oxygen into the oil, removing lighter vapors, separated carbon and sedimentary deposits, vaporizing 130

the remaining oil, and dephlegmating and separating the overhead under a vacuum.

separating the overhead under vacuum, and withdrawing from the dephlegmating and separating zone a plurality of fractions of predetermined quality.

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5	presence of one or more colloiddally dispersed solid adsorbent addition agents having the catalytic properties of colloidal bentonite under super-atmospheric pressure, by heating to a temperature between 500° and 850° F., both inclusive, circulating and agitating under such pressure and while so heated removing lighter vapors, separated carbon and sedimentary deposits, vaporizing the remaining oil with the dispersion in it of the additional adsorptive addition agents, and dephlegmating and separating the overhead under a vacuum.	70
10		75
15		80
20	3. The process of producing lubricating oil which comprises treating oil stock in the presence of one or more colloiddally dispersed solid adsorbent addition agents having the catalytic properties of colloidal bentonite under super-atmospheric pressure, by heating to a temperature between 500° and 850° F., both inclusive, circulating and agitating under such pressure and while so heated and at the same time forcing air or oxygen into the oil, removing lighter vapors, separated carbon and sedimentary deposits, vaporizing the remaining oil with the dispersion in it of the additional adsorptive addition agents, and dephlegmating and separating the overhead under a vacuum.	85
25		90
30		95
35	4. The process of producing lubricating oils which comprises, as a step in their manufacture, pretreatment of the oil stock in the presence of solid colloiddally dispersed adsorptive agent under superatmospheric pressure with heating to between 500° and 850° F., while agitating and circulating for the purpose of removing, transforming and vaporizing compounds and fractions which, in use of the oil, cause deterioration and objectionable deposits, distilling and vaporizing the oil in the presence of the dispersed adsorbent addition agent in a vacuum at a temperature below that at which substantial cracking will take place, and dephlegmating and separating the overhead under vacuum.	100
40		105
45		110
50	5. The process of producing lubricating oils which comprises, as a step in their manufacture, pretreatment of the oil stock in the presence of solid colloiddally dispersed adsorptive agent under superatmospheric pressure with heating to between 500° and 850° F., while agitating and circulating for the purpose of removing, transforming and vaporizing compounds and fractions, which, in use of the oil, cause deterioration and objectionable deposits, distilling and vaporizing the oil in the presence of the dispersed adsorbent addition agent in a vacuum at a temperature below that at which substantial cracking will take place, dephlegmating and	115
55		120
60		125
65		130