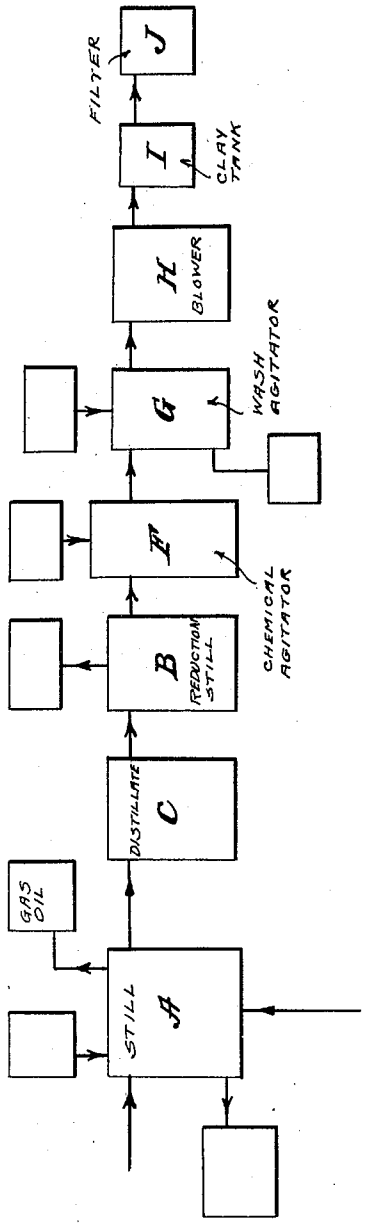
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PROCESS OF MAKING LUBRICATING OIL

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PROCESS OF MAKING LUBRICATING OIL

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The present invention relates to a process of manufacturing lubricating oils, and has particular application to the production of oils suitable for lubrication from asphalt or naphthene base crudes.

It is an object of the invention to utilize as much of the heavy fractions as possible, since it has been established that the so-called bottoms are highly valuable for lubricating oils.

To this end, the invention contemplates but a single complete distillation of the crude thereby materially lessening the apparatus employed and shortening the time of operation, both as regards the production of the distillate and its subsequent chemical and mechanical treatment.

The generally accepted method of refining an asphalt or naphthene base crude for the production of lubricating oils consists in distilling the crude and separating the desired fractions. The various lubricating fractions are then redistilled in order to obtain the desired flash and viscosity. The lubricating fractions obtained during the process of redistilling that do not have the desired flash and viscosity are collected and further refined by reducing or blending with other products. On the redistillation of the lubricating distillate the generally accepted practice is to add a weak caustic solution to the lubricating distillate during the process of the second distillation. After the lubricating distillate is redistilled and the desired fractions obtained, the product is then transferred to the agitator for chemical treatment. The chemical treatment consists of first agitating the distillate with a fixed charge of 66° Bé., sulphuric acid. The purpose of the weak acid treatment is to remove any water that is contained within the distillate. After the acid and the acid sludge produced from this treatment are removed, the charge is further treated with a much stronger acid, usually a powerful acid as a fuming sulphuric acid of substantially 98% concentration, and upon the removal of the acid sludge produced from this treatment, the charge is then transferred to a wash agitator where the remaining acid held by the distillate is neutralized with a weak caustic soda or a soda ash solution. After the excess caustic solution is removed, the distillate is transferred to a blow bright tank where the traces of moisture held by the distillate is removed by blowing with air.

This process is objectionable, because in the first distillation, a base of 5 to 10% remains, which base is generally employed as fuel oil, and on redistillation of the lubricating distillate, it is necessary to leave a base of from 5 to 8% which is utilized also for fuel purposes. Thus, anywhere from 10 to 20% of the crude and particularly that part possessing more valuable lubricating qualities is lost.

The figure is a flow sheet illustrating the process.

In this figure, A is the still wherein the crude oil is run, and where agitation with steam in the presence of caustic takes place. The gas oil and other fractions are drawn off and the remaining residue is distilled overhead and collected in the chamber C. From the chamber C, the residue is run into the reduction still B, where the oil is given the desired viscosity and flashpoint, and lighter fractions are removed. The dry distillate is then run into the chemical agitator F, whence it is subjected to acid treatment with either 66 Bé. or stronger usually and preferably the former. The material is then passed to a wash agitator, and may, if desired, be treated with a weak caustic, and the excess caustic is removed and the chemically treated oil is passed to an air blower H. In some cases, the air blower H is not employed, and the oil is passed to a clay treating tank I, and thence to a filter J, from which the oil may be passed continuously to a vacuum apparatus to get out any remaining water.

With the present invention, I have found that the final product of my process is a lubricating oil of relatively light color possessing a distinct greenish fluorescence free from bluish tints, and furthermore, I do not find it necessary to blend my product with a second distillate in order to secure the desired light color. My process is also

characterized by the fact that the unsaturated hydrocarbons can be removed without interference with the cyclic or naphthene ring compounds, and my improved oil is particularly free from the emulsifying constituents preventing any possibility of the emulsification occurring during treatment with chemicals.

As a result of tests conducted with my product, it is found to be stable and highly resistant to heat, due to the fact that the lubricating oil is substantially free from organic acids, resins, sulfo-acids, unsaturated hydrocarbons which are easily oxidized, gums and phenols which are apparently converted into alkaline stable products by the single distillation which I employ, and which are left behind as part of the base.

Aside from the fact that I do not contemplate the usual double distillation, my invention also enables the process to be carried out in its subsequent steps of chemical and mechanical treatment with materially less expense and time, and the resultant product yield in both quantity and quality indicates that the process is highly efficient in practical use.

In carrying out the process, the crude oil to be distilled which may be any usual crude, is charged into a still and agitated as for example, by applying steam at the bottom thereof as is well known. During this agitation, there is added to the oil a weak caustic solution which is preferably gradually introduced. While various caustics may be employed in one test which I have conducted with Mirando crude which is often referred to as a naphthene base crude, I prefer to use substantially one and one-half quarts of sodium hydroxide solution (about 15 Bé.) per barrel of crude.

My preference for employing a caustic such as sodium hydroxide is the result of experiments wherein it was found that such bases as calcium hydroxide, ammonium hydroxide and others of this type are unsatisfactory and have a different action from that of the caustic, making them wholly undesirable.

The oil is then subjected to distillation, and the agitation by means of the steam is continued and lighter fractions consisting of gas oil etc., are removed by over head distillation and transferred to a suitable storage reservoir.

After it has been ascertained that the gas oil content and lighter fractions have been removed, the entire remaining stock is subjected to distillation, with agitation of the material as previously described. This distillation is continued until all of the oil has been distilled over head with the exception of possibly 5% of bottoms.

The small percentage of bottoms is pumped from the still and utilized for fuel purposes.

The entire lubricating fraction is collected and transferred to a tank or still for reducing the oil and obtaining the desired flash and viscosity.

The reduction process comprises heating the oil to remove the lighter fractions which are distilled over head until the remaining base or charge meets the desired test.

In this manner, no redistillation with consequent apparatus extensive heat and loss of time is employed, and the flash and viscosity of the lubricating stock may be easily controlled. For example: a viscosity of 200 Saybolt and flash of 325° Fahrenheit can be obtained with very little reduction. On further reduction with the percentage of the base decreasing in proportion to the amount of lighter products distilled off, it is possible to obtain in this simple manner a base possessing for example, greater viscosity and higher flash to a considerable range, as required.

It will be here noted that the entire base is reduced not only without its redistillation, but also without necessity of blending. When this base has been reduced to the desired viscosity and flash, it is transferred to a suitable agitator for chemical treatment.

At this point, it is thought well to explain that the caustic has been present in the oil through all the operations previously described, and while possibly some of it may have been lost, this has been found not to appreciably effect the operation of the process.

By reason of the presence of caustic in the reduced distillate, I am permitted to employ a weak acid treatment and I prefer to treat the distillate with 66 Bé., sulphuric acid.

Whereas in former processes, an initial treatment with 66 Bé., acid is necessary as a water cutter, I eliminate this step since the distillate delivered to the agitator and chemical tank is substantially dry.

While I employ preferably a weak acid treatment, using a sufficient quantity of acid dependent upon the viscosity and quality of the distillate being treated, I have in some cases used a stronger acid of 98%. However, for most purposes the weaker acid has been completely effective in producing an oil having the desired quality and color.

After the distillate has been subjected to acid treatment and the resultant sludge withdrawn, the distillate is transferred to a suitable wash agitator where the remaining acid held by the distillate is neutralized with a weak solution of caustic or soda ash if found necessary.

It should here be stated that the reduced distillate produced from the distillation of the caustic crude may be more easily treated in the agitator and with less likelihood of

emulsification than is the case with processes heretofore employed.

The excess caustic is then removed and the distillate is transferred to a rather low but large diameter storage tank and blown with air for the purpose of removing the water held by the distillate.

In case a pale oil is desired, the oil is transferred after the alkali treat to a heat exchanger where it is subjected to a temperature sufficient to raise the oil to a point where it will react with a decolorizing agent. The heated oil at such temperature is passed to a mixing tank and a decolorizing clay is added while the mass is agitated and the reaction temperature of the oil is sustained by means of heating elements arranged in the mixing tank. Since reaction takes place at once between the oil and clay, the oil may be immediately filtered so that the process is very rapid and can be conducted with a minimum of complicated apparatus and without any loss of time.

The filter is preferably of a type wherein the deposits can be flushed or sluiced from the chambers by means of water, without requiring dismantling of the filtering apparatus. The employment of such a filter avoids the usual delays incidental to taking down the apparatus to remove the cake and then assembling. By using a filter from which the cake is sluiced off by streams of water, my process is rendered so to speak continuous and the time of operations is materially reduced. A small quantity of water will pass through with the oil and since the oil is at a temperature above boiling, the water content may be easily removed by vacuum treatment.

As previously stated, application of the caustic solution to the crude prior to distillation, permits the use of relatively weak sulphuric acid in the chemical treatment, and whereas without the addition of this caustic, it would be necessary to employ a more powerful acid as fuming sulphuric acid of substantially 98% concentration. When the distillate is treated with 66 Bé., acid and subsequently clay treated, the desired greenish fluorescence is very pronounced while treatment with 98% acid or stronger, does not result in so definite a greenish cast.

The bluish tint usually found in the distillate is entirely absent from my product, and I attribute this to the use of caustic within the crude during the period of distillation, experiments having shown that without the use of caustic, a very pronounced bluish fluorescence is produced. It is further desirable to employ a weak acid treatment in accordance with my process, since I have found that the distillate can be further refined by means of clay without first neutralizing with caustic soda or soda ash solution. This eliminates the so-called alkali treatment, and the consequent time and apparatus required in the usual neutralizing. Of course, if a stronger acid is employed a neutralizing is necessary before clay treating.

In carrying out my process, utilizing the distillate produced from caustic crude, there is a much smaller loss due to sludge and tarry matter commonly called acid sludge. This is due to the fact by my process the unsaturated hydrocarbons are removed without interference with the cyclic or naphthene ring compounds.

The presence of the naphthene ring compounds is obviously a very desirable feature, and is primarily the result of the relatively light acid treatment to which the oil is subjected.

By introducing a small amount of weak caustic into the crude and agitating the mass prior to subjecting the oil to distillation, I find that various organic acids, resins, phenols, sulfo-acids are converted into stable alkaline products by distillation and remain behind as part of the bottoms.

In treating the crude with caustic and then heating and distilling to temperatures as high as 675° Fahrenheit and 700° Fahrenheit, the naphthenic acids and other organic acids are neutralized and as stated, stable alkaline products such as sodium salts are formed by reaction with the nitrogenuous and oxygenated bodies present.

I have tested the product produced in the above manner, and find that the oil is stable at high temperatures. For example, a quantity of the finished oil has been subjected to a temperature of substantially 300° Fahrenheit more or less for a considerable length of time without material change either in color or quality.

It will be understood from my process that the percentage of bottoms which is lost in the single distillation as well as the percentage of sludge which is lost in the acid treatment is very low, and since it has been definitely established that the so-called bottoms obtained during the refining process of a lubricating oil contain the most valuable lubricant, the importance of my improvements can be readily seen.

Furthermore, by using a weak sodium hydroxide solution, or other caustic solution, there seems to be an increased tendency of the oil not to crack at the higher temperatures. This property is very noticeable as extreme low bottoms can be obtained without increasing to a noticeable degree the quantity of cracked products. The use of a weak caustic solution moveover produces water soluble products which are easily removed by washing.

My process is further characterized by the fact that air is excluded so that no oxidized

products such as asphaltine, tarry and gum forming bodies are formed. My improved method of refining produces a much greater yield inasmuch as only one over head distillation is made, and it is only necessary to leave enough bottoms to protect the still from burning or over heating. The bottoms usually produced by the common second distillation are utilized by me as lubricating oil.

If a strong solution of caustic were employed in lieu of the weak caustic of my process, and which is added in small quantities, there would be a considerable loss of the original oil due to an emulsification of the oil. With a strong solution, it would be almost impossible to obtain a clean cut separation of the oil and alkaline solution. Moreover, by using a weak caustic solution, the percentage of objectionable products, such as soaps is reduced to an inappreciable amount.

If a strong caustic solution were used, there is a possibility of the entire charge emulsifying which is difficult to break, often requiring a separate distillation.

With my process employing a weak caustic solution during heating and distillation, there is no loss of crude by reason of separation of oil and caustic liquor. Moreover, no emulsification takes place and there is no loss due to such reaction. I also find that when subjected to further chemical treatment, the possibilities of emulsification of the distillate is reduced to a minimum.

It will be observed that the use of complicated apparatus is reduced since no double distillation is necessary and the neutralizing step in the chemical treatment may be eliminated. Moreover, the initial alkali treatment is carried on without any additional apparatus.

The time of operations for conducting my process is materially reduced, and this, of course, is a very important item.

Both time and apparatus for have their effect in the cost of production and my invention very materially reduces the expense of refining as will be quite clear.

By the use of the improved method of refining lubricating oil, a product having a green cast can be produced, and also a red oil having a green fluorescence can be obtained. There is but a single distillation and no necessity of blending when operating in accordance with my process. Also, it should be borne in mind that the treatment herein described results in a very high yield of excellent quality lubricating oil at a relatively small cost and within a time period which has been much shortened over the present accepted methods.

It will be understood that where the usual processes having for their purpose the production of a green cast lubricating oil, employ artificial means to acquire and accentuate the cast, my process gives a uniform product of green cast for both red and pale oils without further treatment.

Blending of bottoms from cracked fuel with a distillate is one artificial expedient, while the use of bright stock is another.

Such additional treatments are enormously expensive from the standpoint of operation, but have heretofore been necessary to give the oil the desired color and render it marketable.

My process obviates this and not only shortens materially time of operation and cost of blending material, but actually utilizes the major part of the bottoms which are considered the valuable constitutent and were hertofore generally unused in the direct refining treatment. By using a caustic crude and following the steps heretofore outlined, my process gives a clear product having a percepticle and stable greenish cast.

I claim:

1. The process of refining oil which comprises introducing the oil into a still, adding a small quantity of dilute, caustic, alkali solution thereto, distilling off the lighter fractions, continuing distillation of the remaining charge of caustic and oil until substantially five percent of bottoms remain, reducing said last mentioned distillate by heating to remove lighter fractions only, subjecting the reduced distillate to a chemical treatment with acid of less than ninety-eight percent concentration, removing the sludge, treating with absorbent clay to decolorize the oil, filtering the oil, and subjecting the oil to a brightening treatment.

2. The process of refining oil which comprises introducing the oil into a still, adding a small quantiy of dilute, caustic, alkali solution thereto, distilling off the lighter fractions, continuing distillation of the remaining charge of caustic and oil until substantially five percent of bottoms remain, reducing said last mentioned distillate by heating to remove lighter fractions only, subjecting the reduced distillate to a chemical treatment with acid of less than ninety-eight percent concentration, removing the sludge, heating the distillate to reaction temperature with a decolorizing agent, introducing a decolorizing agent into the distillate when the latter is at such a temperature that the agent will immediately react, continuously filtering to separate the oil and agent, and subjecting the filtered distillate, while still in heated condition, to a vacuum treatment.

In testimony whereof I have hereunto set my hand.

WILLIAM T. BRYANT.