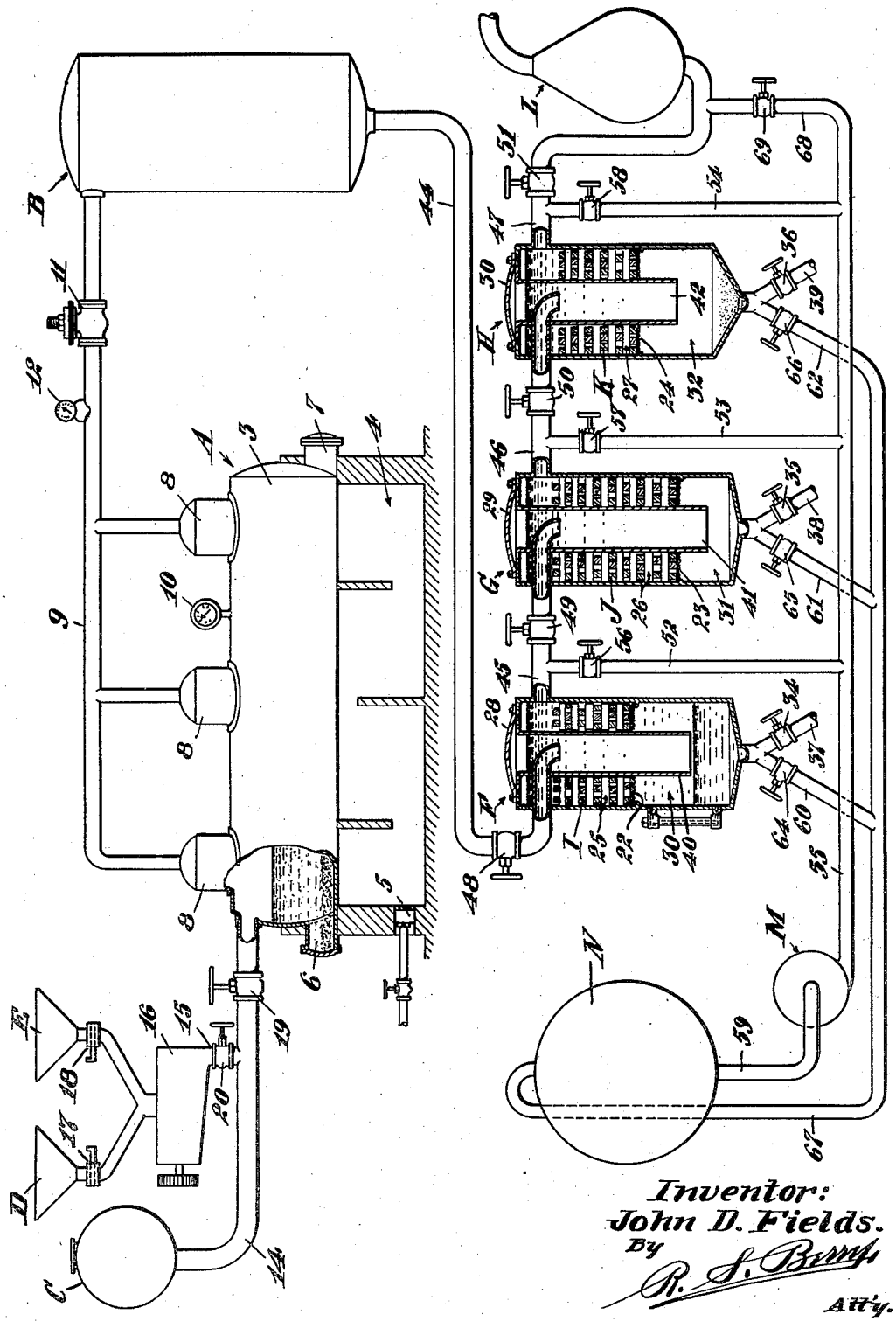
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METHOD OF REFINING HYDROCARBONS

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METHOD OF REFINING HYDROCARBONS

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This invention relates to a method of refining hydrocarbons and particularly pertains to the treatment of petroleums to effect the production of various products therefrom.

The primary object of the invention is to provide a process whereby any petroleum may be treated to produce any one or more of a number of the products of distillation in a single apparatus at the will of the operator and at the same time effect the production of a hard, dry, commercial coke from the residuum, and also to effect the removal and recovery of resinous and other gummy constituents which result in discoloration of the distillates and the imparting of objectionable odors thereto.

Another object of the invention is to provide a method whereby a hydrocarbon may be distilled to effect its conversion into low boiling fractions and coke, whereby a high percentage of production of low boiling fractions of the hydrocarbon may be obtained and whereby residue carbon will be rendered of such character as to enable its ready removal from the distilling apparatus and also have properties imparted thereto which will render it valuable as a carbon or coke product suitable for use in metallurgical and other operations.

Another object of the invention is to provide a method for purifying the distillates to effect the production of a sweet smelling, water white, stable distillate product of great brilliancy.

Another object is to provide a method of refining petroleum or hydrocarbon distillates whereby resinous compounds and other gummy impurities held in solution in the distillates may be removed without destroying the benzol equivalent and octane number constituents of the distillate, whereby such constituents are conserved so as to produce a high grade product which when utilized, as in combusting it in a motor, will not form gummy or resinous deposits.

Another object is to effect recovery of the removed resinous or gummy impurities in a form to permit of their utilization, such for example as a component of fuel oil, and also in the production of various substances of commercial value.

Another object of the invention is the provision of a method whereby the distillates may be refined without material loss thereof thus enabling the production of a greater volume of the purified product from a given volume of distillates under treatment than is obtained from the usual refining processes.

A highly important feature of the invention resides in dispensing with the use of reactive agents, alkalies, filtrates and other substances which are lost in the treatment of distillates by the usual acid and alkali wash processes and consequently require replenishment at considerable cost, thereby obviating the expense incident to the use of such substances and thus resulting in a process for refining distillates which is of great economic value.

The invention resides generally in distilling a hydrocarbon together with a vegetable substance, with or without the addition of iron ore according to the character of coke product desired, effecting such distillation at predetermined temperatures and pressures according to the fractions desired thereby producing distillates in vapor form, condensing the produced vapors, and finally treating the condensed distillates to remove and recover resinous and other impurities therefrom; the invention further residing in effecting the last named step by subjecting the impurities in the condensed distillates to the action of a catalytic agent of a character such that it will act on the impurities and cause same to separate from the benzol equivalent and octane number components of the distillates in a manner which will permit ready removal of the impurities.

The invention and the bode of accomplishing the objects and advantages as may subsequently appear, further resides in the steps and features hereinafter more fully described and claimed and illustrated by way of example in the accompanying drawing in which the figure is a diagrammatic view of a refining apparatus embodying associated instrumentalities employed in producing the results sought.

Referring to the drawing A indicates generally a still for initially distilling the petroleum to form distillates and coke, and which embodies a tank 3 arranged over a fire box 4 in which heat is generated, as by an oil burner 5. The tank is fitted at its ends with normally closed man-holes 6 and 7 for clean out purposes and through which coke residuum is removed, and is fitted with one or more domes 8 connecting with a vapor discharge conduit 9 leading to a condenser B. A pressure gauge 10 is provided on the tank and a pressure relief valve 11 is provided in the conduit 9, which pressure relief valve is adjustable to regulate the pressure at which vapors flow to the condenser from the still in a usual manner. A temperature indicator 12 is mounted on the conduit 9 between the still and the pressure relief valve and adjacent the latter to indicate the temperature of vapors within the conduit. A feed pipe 14 leads to the tank from a reservoir C or other suitable source of hydrocarbon or petroleum supply, through which the materials to be treated are directed to the still to charge the latter as occasion requires, and connecting with the feed pipe 14 is a conduit 15 leading from a mixer 16 to which comminuted vegetable matter is delivered from a hopper D together with or independently of finely comminuted iron ore directed to the mixer 16 from a hopper E; the feed of materials from

the hoppers D and E being controlled by valves or gates 17 and 18. The feed pipes 14 and 15 are fitted with cut-off valves 19 and 20.

The apparatus as shown in the drawing also embodies a series of catalyzer containing tanks F, G and H within which are mounted apertured catalyzer supports 22, 23 and 24 which are spaced from the bottoms of the tanks and are here shown as composed of wire netting. The spaces 25, 26 and 27 above the supports 22, 23 and 24 constitute catalyzing chambers within which are contained catalytic bodies I, J and K carried on the supports, which bodies are formed of or include alkali metal, such as sodium, potassium and lithium.

The catalytic bodies are so shaped and so arranged within the catalyzing chambers as to permit the flow of distillates through said chambers yet cause the flowing distillates to be deflected into numerous small streams which flow through the interstices between adjacent catalytic bodies in such manner that by the time a volume of distillates has passed through a catalyzing chamber the major portion thereof will have been brought into superficial contact with or in close proximity to a considerable area of the surfaces of the catalytic bodies. For this purpose the catalyzers may be shaped in the form of spheres, blocks or bars. They are placed within the tanks as by introducing them through man-holes with which the latter are equipped and which are normally closed by covers, as indicated at 28, 29 and 30.

The spaces 31, 32 and 33 beneath the catalyzer supports 22, 23 and 24 constitute settling chambers in which water and other separable foreign contents of the distillates are collected and drawn off from time to time through drain-cocks 34, 35 and 36 fitted in drain pipes 37, 38 and 39 leading from the bottom portions of the tanks to suitable points of discharge. The tanks are fitted with depending tubular baffles 40, 41 and 42 which lead downwardly from the upper ends of the tanks through the catalyzer supports and open to the settling chambers beneath the supports in spaced relation to the lower ends of the tanks.

A conduit 44 leads from the discharge end of the condenser B into the upper end portion of the baffle 40 in tank F; a conduit 45 leads from the upper end portion of tank F into the upper end portion of baffle 41 in tank G; a conduit 46 leads from the upper end portion of tank G to the upper end portion of baffle 42 in tank H; and a conduit 47 leads from the upper end portion of tank H to a finishing still L. The conduits 44, 45, 46 and 47 are each fitted with cut-off valves 48, 49, 50 and 51 respectively, and leading from the conduits 45, 46 and 47 are pipes 52, 53 and 54 leading to a conduit 55 connecting with the discharge side of a pump M. The pipes 52, 53 and 54 are fitted with cut-off valves 56, 57 and 58. The intake side of the pump M connects through a conduit 59 with a tank N containing a mixture of benzine and alcohol.

Leading from the lower end portions of the tanks F, G and H are pipes 60, 61 and 62 fitted with cut-off valves 64, 65 and 66, which pipes connect with a pipe 67 leading to the upper portion of the tank N. The pump M and its connections affords a means for circulating a benzol and alcohol mixture through the tanks F, G and H separately or collective when effecting cleansing thereof, as will be later described.

A conduit 68 fitted with a cut-off valve 69 leads from conduit 55 to the conduit 47 through which the solvent may be directed from the tank N to the still L.

In carrying out the invention by means of the apparatus here shown, a liquid hydrocarbon is delivered to the still A through the conduit 14 from the reservoir C together with a quantity of ground or comminuted vegetable matter to which vegetable matter is added, where required, a quantity of finely comminuted iron ore; the iron ore being added where it is desired to produce a ferric coke of hard character and being omitted where it is desired to produce a soft, dry coke.

As is well known there is a difference in the quality and character of the two kinds of coke referred to, and that such cokes have distinctive properties which render them suitable for use for different purposes, for example, soft coke is more valuable than a hard coke in the manufacture of carbon articles, and as a fuel in certain types of stoves or furnaces, while the hard coke with an iron content is useful in blast furnaces and metallurgical operations where the use of soft coke is not practical.

The hydrocarbon to be treated may be of any character; the invention, however, being particularly applicable to the treatment of petroleum of either asphalt or paraffin base having a high boiling point, for example, of approximately 650 degrees Fahrenheit.

The vegetable matter employed is preferably of such character as to contain alcohol, acetic acid, aceton, phenols and terpenes. An example of such vegetable matter suitable for the purpose is sawdust, kelp, beet pulp, sugar cane, and other substances of similar nature, but any vegetable matter capable of being converted into charcoal may be utilized. Vegetable matter in a green or natural state is preferably employed.

The iron ore may consist of any ore which is subject to being reduced to metallic iron, and before being added to the other substances is comminuted to such state of pulverization or fineness that when added to said substances will be held in suspension by the vegetable matter. Iron ore reduced to a fineness of from 150 to 250 mesh has been found satisfactory.

The vegetable matter, iron ore and hydrocarbon may be combined in various proportions depending on the characteristics of said substances and the nature of the products thereof desired, but the hydrocarbon is employed in sufficient quantity relative to the quantity of the vegetable matter as to saturate the latter. A specific proportion of the substances found to be satisfactory is 10 parts of vegetable matter, 10 parts of iron ore and 80 parts of a liquid hydrocarbon.

The mixture of substances placed in the still is subjected to the combined action of a temperature and pressure sufficient to effect distillation of the vegetable matter, and thereby drive off essential oils and acids and other vaporized constituents; to reduce the iron ore to metallic iron; to separate the low boiling fractions from the hydrocarbon, and to reduce the residuum to a dry coke. A temperature of from 350 to 650 degrees Fahrenheit has been found highly satisfactory, although higher temperatures may be employed as where heavier fractions are to be produced; it being evident that the higher the temperature to which the materials are subjected in the still the greater separation of the heavier fractions from the hydrocarbon under treatment will result.

While a wide range of pressures may be employed in the still during the distilling operation, it has been found in practice that by subjecting the material under treatment to a pressure of approximately 100 pounds with a temperature of ap-

proximately 650 degrees Fahrenheit a maximum of carbon is produced with a maximum of low boiling fractions, although variations of the pressure and temperature are resorted to as occasion may require.

The pressures in the still A are indicated by the pressure gauge 10 and the temperatures of the distillates are determined by the temperature indicator 12. The desired pressure and variations thereof are obtained by adjusting the pressure control valve 11, and the desired temperature and variations thereof are obtained by controlling the heat generated in the fire box 4 as by regulating the burner 5. A reduction of temperature in the still may be effected by feeding additional liquid hydrocarbon with or without the vegetable matter to the charge contained in the still.

A particularly effective mode of operating the still in treating petroleum with its added content is to initially heat the contents of the still until the distillate vapors, which pass off through the conduit 9, have a temperature of 400 degrees Fahrenheit or thereabout; this temperature being maintained for a suitable period to distil off the required amount of the gasoline content of the hydrocarbon under treatment. The pressure control valve is then set to open at 100 pounds pressure and the temperature increased to 580 degrees or thereabout to effect conversion of high boiling fractions into low boiling fractions. The temperature is then gradually raised to 650 degrees or thereabout, which temperature is maintained until all high boiling fractions are vaporized, and may be continued until the residuum in the still is dried out, but the temperature may be increased to say 750 degrees where it is desired to hasten the drying action. At some point after increasing the temperature to 650 degrees or thereabout, that is when the volatiles are sufficiently driven off, the pressure control valve is adjusted to open under normal pressures and is maintained open so as to relieve the still of internal pressures during the drying out operation. An important feature of the invention resides in the fact that the distillation may be completed at temperatures up to and not exceeding 650 degrees Fahrenheit or thereabout, due to the volatile content of the vegetable matter, thus reducing the amount of heat required during distillation under pressure in converting petroleum into gasoline and other distillates over other methods of distillation of petroleum now generally in use.

About ninety-four per cent of the original oil charge is volatilized and passes from the still at 650 degrees Fahrenheit or thereabout, leaving about six per cent of residuum oil in the still together with a quantity of residue charcoal, which latter results from the heat treatment of the vegetable matter contained in the oil.

This charcoal is formed before the formation of mineral carbon or coke takes place and results in the depositing of a layer of the charcoal on the inner surface of the still before any appreciable amount of mineral carbon or coke is formed in the still so that when such mineral carbon or coke is formed it is deposited on the layer of charcoal and accordingly practically no accumullation of the mineral carbon or coke directly on the metallic walls of the still takes place. Where iron ore is added, the vegetable matter will hold it in suspension so as to prevent its precipitation to the bottom of the mass during the distilling operation, and hardens coke as the latter forms on the walls of the still, thus rendering the formed coke highly conductive of heat so that it will serve to protect the walls of the still against burning out during the final stages of drying out the coke and also serves to hasten the driving off of the final volatiles from the coke residuum. The iron containing coke also acts as a conductor of heat from the bottom of the still throughout the mass under treatment. However, where the ore is not used, the charcoal will in like manner form with mineral carbon a protective coating on the walls of the still.

When the batch under treatment has been subjected to the distillation temperatures as above stated until the desired fractions have been driven off and the charcoal and coke body has been formed the temperature may then be raised to 750 degrees or thereabout if desired so as to more rapidly drive off the residue oil in vapor form and hasten drying of the coke and charcoal residuum as before stated; the resultant vapors on being condensed forming a high grade lubricating oil. This higher temperature is rendered possible by the protective action of the body of charcoal and coke on the walls of the still.

In this manner the petroleum distillates are completely removed and the residuum dried out. This residuum will consist of an admixture of vegetable carbon or charcoal and mineral carbon together with such other solid substances as may result from distillation of the materials under treatment, it including in its composition mineral carbon derived from the petroleum together with charcoal derived from the vegetable matter, and will contain metallic iron derived from the iron ore when such ore is used.

After the drying out stage has been effected the carbon or coke body formed in the still is removed from the latter, as through the man-holes 6 and 7, which operation is readily accomplished by reason of the charcoal layer formed on the walls of the still being easily separable therefrom; the charcoal having no tendency to tenaciously adhere to the metallic surface of the still but only to rest thereon. This is highly advantageous as the mineral carbon resulting from the distillation of petroleum without the use of vegetable matter, as in the common practice, has such affinity for the heated metal of the still, usually iron, as to cause such adhesion of the carbon to the metal as to render its removal very difficult. Furthermore the finished coke product obtained by my process will contain not to exceed one per cent volatile matter whereas ordinary cracking still coke contains eight to eleven per cent volatiles, and accordingly the coke product thus obtained will require no further treatment, but is in commercial form as it comes from the still and is in condition for use. Again, the coke product is hard and firm, with practically no fines and is characterized by a high carbon content. Coke, minus the iron content, can be produced with an average of ninety-five per cent fixed carbon. The coke made according to my process is very low in sulphur content, since the vegetable matter with which the petroleum is distilled converts the sulphur originally in the petroleum into compounds which are removed from the coke residue.

By the addition of the vegetable matter to the petroleum other marked advantages are derived and highly desirable results are attained. For example, when distillation commences, acetic acid is given off by the vegetable matter, which combines with hydrogen sulfide in the oil and thereby effects its removal; methyl and ethyl alcohol

are released; the terpenes or series of essential oils contained in the vegetable matter are polymerized into lighter fractions of ethylene, butylene, pentane, xylene, and other desirable fractions of gasoline including benzine and benzol; the vegetable matter apparently assists in the cracking of the heavier fractions, causing them to be converted into low boiling compounds, at a much lower temperature and at much lower pressures than where vegetable matter is not present.

The vegetable matter also serves a useful purpose in that it absorbs a portion of the oil, consequently during the distilling action, moisture and essential oils in the vegetable matter and the lower boiling fractions of the petroleum are produced together. Furthermore, the charcoal residuum resulting from the decomposition of the vegetable matter in depositing on the bottom of the still insures a greatly desired uniformity of temperature throughout the walls of the still and its oil and vapor content, and which is further enhanced by the addition of the iron ore.

The still is charged with a batch of the substances from time to time, the materials subjected to the desired temperatures and pressures, and the accumulated coke removed and the operations repeated as circumstances may require.

During the distilling operation, the operator by regulating the temperatures and pressures can take off the desired distillates such as gasoline, kerosene, terpenes, engine distillate, and kindred products as well as lubricating oil and paraffin waxes, in vaporous form, which products are delivered to the condenser B and there condensed in the usual manner. By controlling the temperatures and pressures in the still only the distillates, kerosene or gasoline alone may be produced as desired.

The condensed distillates contain certain objectionable impurities which necessitates refinement of the distillates to effect removal of such impurities.

While little is definitely known as to the components of distillates of the character above referred to, particularly as to the exact nature of the undesired impurities formed therein, it is generally recognized that such impurities are of resinous and gummy character and include the olefin hydrocarbons embracing mono-olefins, the di-olefins or poly-olefins; sulfur compounds; nitrogen bases; and acid compounds. It has heretofore been the general practice to effect removal of the impurities, whatever their nature may be, by first treating the distillates with sulfuric or other acids; then water washing the acid treated distillates to remove the larger part of the acids that remain in the distillates after the sludge has been allowed to settle; washing the distillates with an alkali wash embodying caustic soda, sodium carbonate, or sodium plumbite, to effect removal of impurities and to neutralize acids; then passing the distillates through a filter of clay or diatomaceous earth, and finally redistilling the distillates.

This practice results in a large waste of the distillates under treatment caused in part by evaporation of the lighter fractions during handling thereof and also results in the loss of the removed impurities which by reason of the acid and alkali treatments are rendered unfit for use, and further entails the loss of the acids, alkalies, and filter media employed in the process. Furthermore, the acid acts to destroy the benzol equivalent and octane number components of the distillates, which if preserved in the refined product would render it of higher grade and of superior quality particularly in the case of gasoline to be used as motor fuel as the benzol equivalents and octane numbers impart to the product greatly desired anti-knock properties.

The distillates while in liquid form are directed through conduit 44 into the upper portion of tank F, being delivered into the upper end portion of the tubular baffle 40 and directed downwardly through the latter into the lower portion of the tank from whence the distillates flow upwardly through the interstices between the catalytic bodies I to discharge through conduit 45 leading from the upper end portion of the tank F.

Water content of the distillates collects in the lower portion of tank F beneath the catalyzers I as indicated at 30 to be drawn off as required through the drain-cock 34, the water level being indicated by a gauge with which the tank F is equipped.

The distillates flowing through conduit 45 are fed into the upper portion of the tubular baffle 41 in tank G and are thence directed through the spaces between the catalyzers J in the latter in the manner described with reference to tank F; the distillates then being delivered from tank G through conduit 46 into the tubular baffle 42 in tank H where the distillates are directed through spaces between the catalyzers K in said tank in a like manner and passed to discharge through the conduit 47 leading from the upper end of tank H.

In this fashion the distillates are directed through any suitable number of tanks as occasion may require to subject the resinous and other gummy impurities of the distillates to the action of the catalyzers a sufficient length of time to effect coagulation of such impurities into a flocculent form; the duration of time being varied according to the character of the distillates under treatment.

In practice it has been found that by maintaining the liquid distillates in juxtaposition with the catalyzers for a period of from three to five hours at ordinary temperatures of the liquid will give highly satisfactory results.

The coagulated or flocculent impurities resulting from the action of the catalyzers collect in part on the catalyzer bodies, and a portion of such impurities are suspended in the distillates a major part of which settles in the tanks G and H and are separated from the distillates in these tanks by drawing off through the drain-cocks 35 to 36 from time to time as occasion may require together with such other separable foreign matter as may accumulate in the settling chambers.

Such portions of the flocculent impurities as do not settle and remain in suspension in the distillates are removed from the distillates by redistilling the latter in the still L to which the distillates are delivered from the end tank H through the conduit 47, or may, if desired, be recovered by filtration of the treated distillates in a usual manner. While I do not wish to bind myself to any particular theory regarding the action of the alkali metal used in connection with the removal of impurities from the hydrocarbon oil, nevertheless it should be stated in the interest of clarity that the alkali metal probably acts to accelerate a condensing action of the impurities in the hydrocarbon, some of which impurities adhere to the surface of the alkali metal while other portions thereof are merely

flocculated and remain in suspension. In other words, the alkali metal merely hastens an action that would undoubtedly occur if sufficient time were allowed, and hence I have referred to the action of the alkali metal as a catalytic action.

The distillates after having been treated in the tanks are preferably redistilled, but not necessarily so; the subsequent treatment of the distillates depending on the character of the treated distillates and the results desired. For example, fresh run gasoline does not ordinarily require redistilling but in some cases may be benefitted thereby. Where the treated distillates embrace light and heavy fractions they are redistilled to separate the distillates into desired fractions such as gasoline, engine distillate, and kerosene.

The catalyzing apparatus as here shown is designed for continuous operation, that is, for a continuous run until conditions necessitate a shut down, and is also designed to facilitate a cleaning out operation to effect recovery of the coagulated impurities as will presently be described, but manifestly any suitable catalyzing apparatus may be employed to carry out the process.

In the course of a protracted run of the distillates through the catalyzing chambers the catalyzers will become coated with a gummy resinous mass consisting of the impurities before referred to, which coating while not interfering with the catalytic action must be removed in order to obviate clogging of the catalyzing tanks and also to effect recovery of the substances constituting such coating. This is accomplished by discontinuing flow of the distillates through the catalyzing tanks, draining the fluids from the tanks, then filling the tanks with a solvent of the separated impurities. For this purpose, benzol is used as a solvent to which is added a quantity of alcohol for the purpose of activating the catalyzers. A volume of alcohol of from five to ten per cent of that of the benzine is ordinarily employed, but any suitable proportion of alcohol may be used as occasion may require.

The solvent is directed through one or more of the catalyzing tanks from the tank N by means of the pump M and the pipes leading therefrom to the catalyzing tanks; the previously closed valves in said pipes being opened and the previously open valves in the several conduits communicating with the catalyzing tanks being closed according to the desired direction of flow of the solvent. For example, on closing the valves 48 and 49 and opening valves 56 and 64 and operating the pump M the solvent will be directed through the tank F, or by closing valves 49 and 50 and opening valves 57 and 65 the solvent will be directed through tank G, or by closing valves 50 and 51 and opening valves 58 and 66 the solvent will be directed through the tank H.

To direct the solvent through the series of tanks the valves 48 and 51 are closed and valves 53 and 64 opened while valves 56, 57, 65 and 66 are maintained closed. When it is desired to redistill the solvent it is directed from the tank N to the still L through conduit 68 on opening the valve 69.

The coated catalyzers are maintained submerged in the benzol-alcohol bath a sufficient length of time to effect dissolving of the coating of impurities, and the bath is removed from the tanks with the dissolved impurities held in solution therein and is distilled in such manner as to effect separation and recovery of the benzol, alcohol and the previously dissolved substances contained therein.

By employing alcohol in the benzol solvent an action is had on the alkali metal bodies such as to hasten the formation of the flocculent gummy resinous matter and lessens its tendency to cling to the surfaces of the bodies, and also renders the separation of the coating from the bodies more rapid and thorough. It is therefore desirable to subject the alkali metal bodies to an alcohol containing bath before initially employing them in the treatment of the distillates.

After having thus washed the catalyzers and cleansed the tanks of the solvent, the apparatus is conditioned in readiness for another run of the distillates to be treated.

The removed impurities are of resinous character and as recovered are in the form of a gum or wax, which substance possesses valuable qualities and may be employed in various uses, such for example as an added constituent to fuel oil, and as a wax for use in the manufacture of candles.

An important feature of the invention resides in the fact that the catalytic action effected by the alkali metal can be carried on at normal atmospheric temperatures and at the ordinary temperatures of the liquid distillates and hence does not require the generation and application of extraneous heat, and also that the action may be carried on at normal pressures.

I claim:

1. The method of treating petroleum oil composed in large part at least of fractions less readily volatile than gasoline, which comprises mixing with such oil a relatively small percentage of a finely divided carbonizable cellulosic vegetable material, the vegetable material being only in such an amount as to form a readily flowable mass; heating such mixture under a superatmospheric pressure, to a temperature sufficient to carbonize the vegetable material but not over 750° C., whereby carbonization of the said vegetable matter is effected, and cracking and distillation of said oil is produced; and condensing the oil vapors, and refining said oil distillate by contacting the same with an alkali metal in the metallic state, while cool, until desulphurization, decolorization and degumming of said oil distillate are sufficiently accomplished.

2. The method of treating petroleum oil composed in large part at least of fractions less readily volatile than gasoline, which comprises mixing with such oil a relatively small percentage of a finely divided carbonizable cellulosic vegetable material together with a small percentage of iron ore, the said vegetable material and iron ore being only in such an amount as to form a readily flowable mass; heating such mixture under a superatmospheric pressure, to a temperature sufficient to carbonize the vegetable material but not over 750° C., whereby carbonization of the said vegetable matter is effected, and cracking and distillation of said oil is produced; and condensing the oil vapors, and refining said oil distillate by contacting the same with an alkali metal in the metallic state, while cool, until desulphurization, decolorization and degumming of said oil distillate are sufficiently accomplished.

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