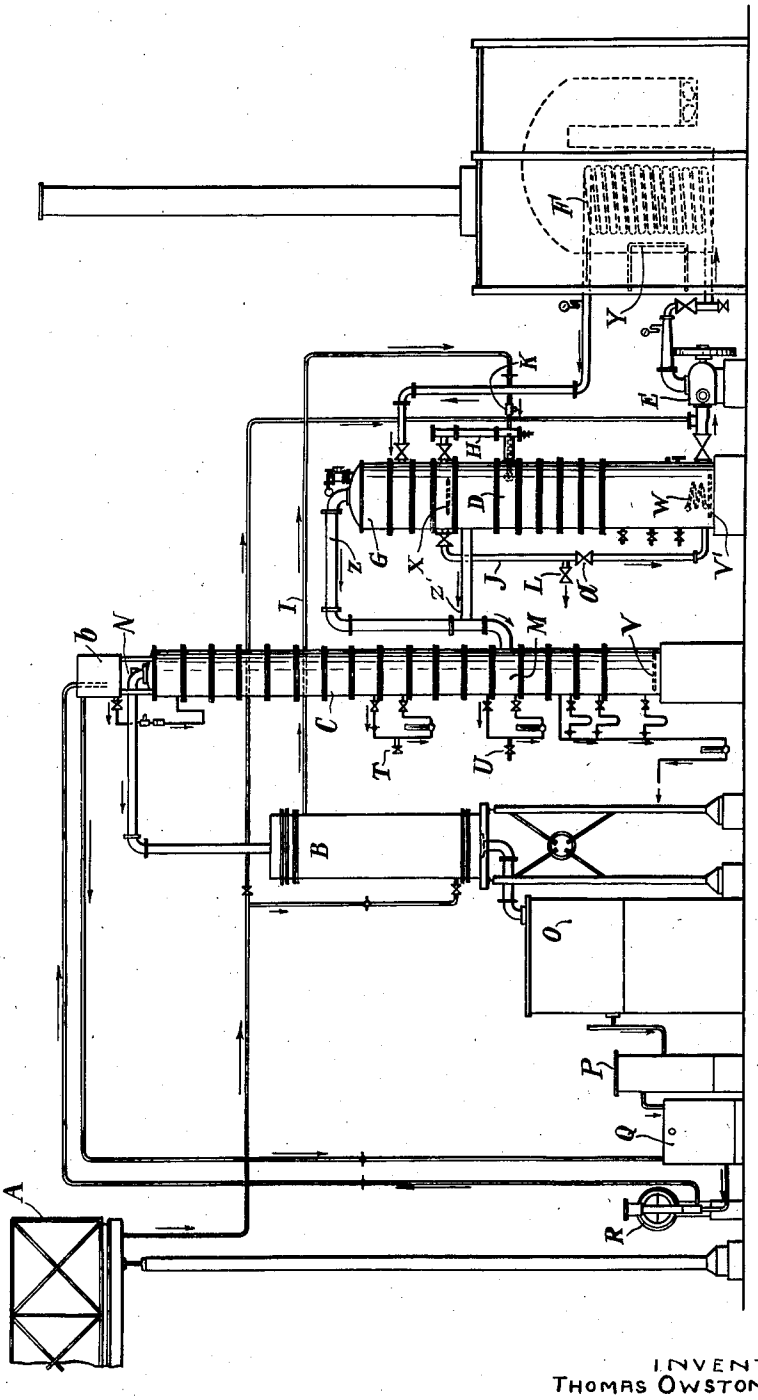
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PROCESS INVOLVING THE DISTILLATION AND FRACTIONATION OF A MIXTURE COMPRISING HYDROCARBON COMPOUNDS INCLUDING VOLATILE AND NONVOLATILE CONSTITUENTS

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INVENTOR:
THOMAS OWSTON WILTON

BY: Thomas E. Boyce

ATTORNEY

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PROCESS INVOLVING THE DISTILLATION AND FRACTIONATION OF A MIXTURE COMPRISING HYDROCARBON COMPOUNDS INCLUDING VOLATILE AND NON-VOLATILE CONSTITUENTS

Thomas Owston Wilton, London, England

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This invention relates to processes for the distillation and fractionation of mixtures comprising non-volatile constituents and volatile constituents, including a plurality of hydrocarbon compounds vaporizable at different boiling points, and it is an object of the invention to continuously separate all of the volatile constituents from the non-volatile constituents and fractionate and condense the volatile constituents as they are separated without intermediate condensation and revaporization

For the sake of illustration the process will be described hereafter in connection with the distillation of tar.

According to the invention the tar, after being deprived of some or most of its low boiling point volatile constituents by being mixed with hot materials having already gone through the process cycle, is caused to circulate through a coil still with a speed which is independent of the throughput of the plant, in this manner avoiding coking up of the coil still, the materials issuing from the still passing through a vapour box where more of the vaporised constituents separate, the pitch residue and the non-vaporised heavier constituents being sent into a mixer column which they enter together with any added increments of preheated new stock to be treated, the entry of the latter being thermostatically controlled by the temperature of the finished product, the mixture of pitch, non vaporised heavier constituents and additions of new stock being again circulated through the coil still whereby a continuous cycle is maintained, the vapours of distillation from the vapour box and from the mixer column being led without condensation to a fractionating column from which the lighter fraction is conveyed to a heat exchanger, where the new stock being fed to the vaporizer is preheated, the said fractions being then condensed, a portion of the light oil fraction being returned to the head of the fractionating column under thermostatic control.

The process is carried out with the assistance of superheated steam introduced by perforated steam pipes at the bottom of the fractionating column, but the pipes may be dispensed with by passing instead the distilled vapours through a superheater for vapour superheating. The vapour box and mixer column are also similarly provided with perforated steam pipes, the contents of the mixer column being warmed up, at the commencement of the process for example, by a superheated steam coil.

In large plants two coils may be used, one to assist in the preheating of stock to be treated and the other to complete the distillation as already described.

Referring to the drawing, the figure is a diagrammatic view illustrating, as an example, an arrangement of plant suitable for carrying out the process according to the invention, which for the sake of illustration, will be supposed to be used for distilling tar and assumed to start from the cold.

The tar flows from a feed tank A by means of a pipe to a heat exchanger B where (when the whole plant is operating) the vapours from a fractionating column C impart some of their heat to the incoming new stock. The new stock flows from the heat exchanger B by means of a pipe I to a mixer column D forming the lower portion of a vapour column, the upper portion of which is referred to as the vapour box G. The admission of new stock at this point is regulated by a thermostat, controlled by the temperature of the finished product.

When starting from cold, the additions of new stock, falling down inclined weir plates provided in the mixer column D, forms a body of tar at the bottom of the column, and is pumped by means of the tar feed pump E through a coil still F and fed to a vapour box G at the top of, and forming part of, the mixer column D. The vapours formed in the vapour box are discharged by a pipe Z at the top thereof, and proceed to the fractionating column at M while the residue or pitch in the vapour box G travels to the bottom of the vapour box over weir plates in the latter, and thence proceed to the mixer column D (which is integral with said vapour box) by means of two pipes J and H opening respectively at the upper part and at the lower part of the mixer column D. The pitch in the pipe J proceeds from the vapour box to the base of the mixer column D and is controlled by means of a cock *a* whereby the throughput of the plant is regulated the said cock being so adjusted as to maintain the quantity of pitch flowing to the bottom of the mixer column slightly in excess of that required for maximum throughput.

The pitch in the pipe H proceeds from the vapour box to the upper part of the mixer column D at a point where the preheated tar to be introduced also enters the column through a thermostat controlled valve K, which is automatically opened when a predetermined temperature prevails in the mixer column, so as to permit more tar to be admitted thereto. The added

tar and pretreated pitch entering the mixing column at this point mix on weir plates and the heat of the pitch vaporises some of the constituents of low boiling point of the tar, so that it is dehydrated and partly distilled on its travel down the mixer column, the vapors being discharged through the pipe Z' and thence to the fractionating column C at point M, and the mixture of crude, partly distilled and dehydrated tar and pitch collecting at the bottom of the column D being again circulated through the coil still and vapour box, by means of the tar feed pump E, in the manner already described, the pitch residue becoming hard pitch and the vapours of the remaining volatile constituents separating into the vapour box.

As the pump E is preferably designed for circulating a quantity of liquid which is ten times the normal output of the plant for the smaller installations, nine-tenths of the circulating material will flow into the upper part of the column to be used for dehydration. The temperature, rising continuously owing to the heat applied at the coil still F, reaches a complete distillation temperature of from 300 to 400° C. in the vapour box G. At this temperature pitch will flow down the pipes J and H from the vapour box and all the vapours generated in the vapour box and in the mixer column during the distillation to pitch are conducted into the fractionating column C and enter the column together at a point M near the bottom section.

When the level of finished residue, here pitch, in the mixer column D has reached a definite height, owing to a sufficient quantity of pitch flowing down the lower entry pipe J, the pitch overflows out of a valve-controlled pitch outlet L provided on that pipe, only pitch being discharged. This may be automatically discharged without any connection between the top and bottom of the mixer column. The plant is so operated that no pitch discharge takes place until the temperature in the mixer column is such that the pitch discharged is a finished product suited in every way to requirements. This is obtained by first testing the pitch at the pitch outlet, and setting the thermostat in the mixer column to open at the temperature of distillation which is required. Directly this temperature is reached, an admission of tar is allowed through the tar admission pipe, and this raises the level of pitch and partly dehydrated tar in the mixer column, and causes the pitch to overflow by gravity. The vapours entering the fractionating column at M condense and form bubbling layers of condensates on the trays of the column, the vapours passing over at N at the top of the column proceeding through the heat exchanger B and away to a condenser O. From the condenser O the condensates pass to a separator P where the water and oils are automatically separated the light oil running into a light oil receiving tank at Q. A thermostat is provided at N, at the top of the fractionating column C, which thermostat can be set to allow vapours at any temperature below a given point to be passed over through the heat exchanger B and into condensers. In order to keep this temperature constant, a reflux pump R is provided which returns a certain amount of light oil from the light oil receiving tank Q back to the fractionating column C at the top section N thereof through a light oil return tank *b* placed above the column. Or the pump can be avoided by disposing the condenser O and separator P, or a second condenser and separator, at the top of the fractionating column, and providing a gravity return to the latter. The thermostat controls the quantity returned, and thus controls the temperature at the top of the fractionating column to any degree required, this temperature being fixed and the temperature at the point of entry of the vapours into the column being that corresponding to the complete distillation of pitch. The boiling point of the condensates on the trays between these two points decreases in proportion to their distance above the point M of admission of the vapours. The liquid fractions at any given temperature can be extracted between these points and any required fraction can be obtained at a suitable point such as T or U. Thus at any point a fraction can be obtained by opening a plug cock (e. g. T or U) provided at the side of each section of the column and allowing the liquid from the section to flow away to storage, and at the same time using some of this liquid to reflux back to the column at this point to maintain the selectivity and boiling range of the desired cut. The quantity allowed to flow to the storage tank can be regulated by means of a cock, so that the temperature is kept at the requisite value, in accordance with the fraction required at that point. The bubbling sections below the admission point of the vapours to the fractionating column are arranged so as to secure as pure a residue-fraction as possible, and this is obtained in case of treatment of some oils by the admission of superheated steam at the point V at the lower part of the column. The superheated steam being supplied at a temperature of approximately 350° C. tends to release any lighter products which may be held in suspension in the section of the column below the admission point M of the vapours, driving them upwards and leaving the residue fraction comparatively pure. By this method of separation any number of fractions of any degree of purity can be obtained with fine limits by elaboration of the fractionating column. Very little water is required at the condenser as the heat exchanger has a considerable condensing effect upon the vapours passing over from the column. The whole of the heat of distillation is thus employed in the fractional separation of the products. In the case of some liquids, heat exchangers may be inserted in the circuit of the two pipes from the vapour box. A superheated steam coil W is provided at the bottom of the mixer column which can be used for warming up said column, also a perforated pipe V' for liberation of products by means of open steam at this point. A further perforated steam pipe X is provided at the base of the vapour box for similar purposes, so that hard pitch can be obtained at a lower distillation point by injecting open steam through the perforated steam pipe at the base of the vapour box. A superheated steam coil Y is placed in the brick setting of the coil still, so as to produce superheated steam for the plant, utilising some of the waste heat of the still.

It will be seen that the plant consists in the combination of a tar distilling plant working on the continuous circulation principle as described in British specifications Nos. 307,577, 337,581 and 424,645, with a fractionating column of a known type, and that, by this process, the distillation of the tar to hard pitch and the fractionation of the volatile constituents can be carried out in one operation, without intermediate condensation and reheating.

It will be also seen that in the process de-

scribed above, vapours are collected at at least two points: (1) on entering the mixer column D and (2) in the vapour box at G.

If desired, a pitch tower adapted for use with highly superheated steam and a condenser, may be fitted to the pitch outlet of the mixer column, so that pitch of varying grades of hardness may be obtained without interfering with the working of the fractionating column, by varying the quantity of steam used in the tower. Vapours may still be extracted from the pitch at this point, which constitutes a third point where vapours are collected.

The plant as it is arranged is self-operating. The thermostat arranged as shown admitting the tar to be treated according to the temperatures at which the plant is to be worked does not permit of excessive temperatures. Should the temperature fall below the distilling point, no discharge at L of the finished product would take place. Thus, with a gas fired still the plant is self-operating; and with a hand fired furnace the plant is self-operating and fool-proof, provided the operator fires the still at reasonable intervals.

The pump E circulates material through the coil still, the vapour box, the mixer column, and back again in a closed circuit, and the tar to be treated is admitted in regulated quantities by the thermostat. The outlet or discharge of the plant will depend upon the level and the quantity of finished products in the mixer D. The faster the pump E is worked the greater will be the quantity of distillates which will be discharged from the mixing column D through pipe Z' and the less will be the amount of vapours which will be discharged from the vapour box G through the pipe Z.

When pitch according to a particular specification is not obtained by straight distillation, due either to specific gravity, viscosity or free carbon, this can be corrected by means of air blowing for effecting polymerization of the constituent hydrocarbons in the tar. A small air compressor or blower, with or without an air heater fitted in the pipe still setting, is fitted to supply a quantity of hot air to the point X in the vapour box, and the air and vapour carried off passes through the condenser, and the condensates are sent back to the column, while the circulated pitch becomes oxidised and thickens, and its carbon contents and viscosity increases. Alternatively, air blowing may be performed after the pitch is discharged at the discharge valve L. This system can also be used when bitumen is added to the column, and the resultant viscosity and free carbon adjusted to very wide ranges.

The revivification of wash oils can also be carried out by the process according to the invention. When this oil has been passed through a benzol plant a number of times, and it has become charged with gums and other heavy oils which cannot be easily removed by steam distillation; this oil can be completely distilled off by the process, without driving off any vapours, and the residues run away to the tar well or used for burning on the fires.

Debenzolising gas oil can also be carried out by this process, but this is only of advantage where revivification of wash oil, with, possibly, tar dehydration is contemplated.

When bitumen mixtures or emulsions are required, the process can be easily adapted for this purpose. In the case of bitumen mixtures, a bitumen melter is installed in the brickwork setting of the coil still, so that the waste heat at from 400° C.–500° C. circulates around the melter and melts a sufficient quantity of bitumen for a continuous run throughout the day. A small flow indicator is inserted in the pipe leading from the melter, and the bitumen is carried by gravity to a point in the mixer column. Falling down cascade fashion in the mixer column, it becomes mixed with the dehydrated tar on its way to the bottom. The pump, which is circulating the material five or six times around the circuit before it is discharged, thoroughly mixes the tar and bitumen, and the discharge from the plant is an efficient bitumen mixture.

With regard to emulsions, it has been described how bitumen can be added, and, of course, any other liquids can be similarly added. Emulsions can be provided, and, in fact, are partially provided, by the very working of the process. The scheme can be completed by the installation of a small mill at the outlet point of the finished product.

What I claim is:

1. A process involving the distillation and fractionation of a mixture comprising hydrocarbon compounds, including volatile and non-volatile constituents, which consists in continuously circulating partly treated stock and added increments of new stock through apparatus including heating means adapted to raise it to and maintain it at a predetermined temperature such as to vaporize all of the compounds which it is desired to separate from the residue; feeding increments of new stock to the partly treated stock being circulated through the aforesaid apparatus, after the separation of vapors therefrom, whenever the temperature of the treated stock would otherwise be caused to exceed the aforesaid predetermined temperature; mixing the new stock with the highly heated, treated stock to effect an initial volatilization of its lighter constituents; continuously conducting the lighter vapors separated from the new stock and the heavier vapors separated from the re-treated stock away from the residue material and assembling them in a single body of vapors of various compounds each volatile at its own characteristic boiling-point; the vapors in the body being permitted to rise from an admission zone, maintained at an approximately uniform maximum temperature as a result of the constant supply from the aforesaid vaporizing apparatus, to successive zones of higher level and of gradually decreasing temperature, and such of them as may fall below a predetermined minimum temperature being permitted to separate from the main body at the zone of highest level and of lowest temperature; condensing vapors at various zones of the composite body of volatilized compounds to produce distillates of boiling-point characteristics dependent upon the temperature of the vapors at the respective zones of condensation; withdrawing the respective distillates as formed; and providing for an outflow of a part of the residue of the stock treated in the circulating system from which the desired distillates have been extracted at a rate dependent upon that of the introduction of new stock irrespective of the rate of circulation through the heating means.

2. A process for distilling tar, comprising distillable oils and residues, by maintaining a pool of material consisting of a mixture of topped fresh charge and residual materials in the lower portion of a mixing column, circulating the material from said pool through a heating coil and

heating it therein to a high temperature, discharging the heated material from said coil into a separator wherein separation of vapors from residues occurs, withdrawing a stream of residues from said separator and passing it directly into the pool in the lower portion of the mixing column, withdrawing a second stream of residues from said separator, and passing it into the upper part of said mixing column, feeding a fresh charge of tar into the upper part of said mixing column, mixing and flowing the tar and residues downwardly in said column into the said pool in the lower part of the column, releasing vapors from the mixed materials during said downward flow, combining such vapors with vapors released in the separator, and fractionating the combined vapors.

THOMAS OWSTON WILTON.