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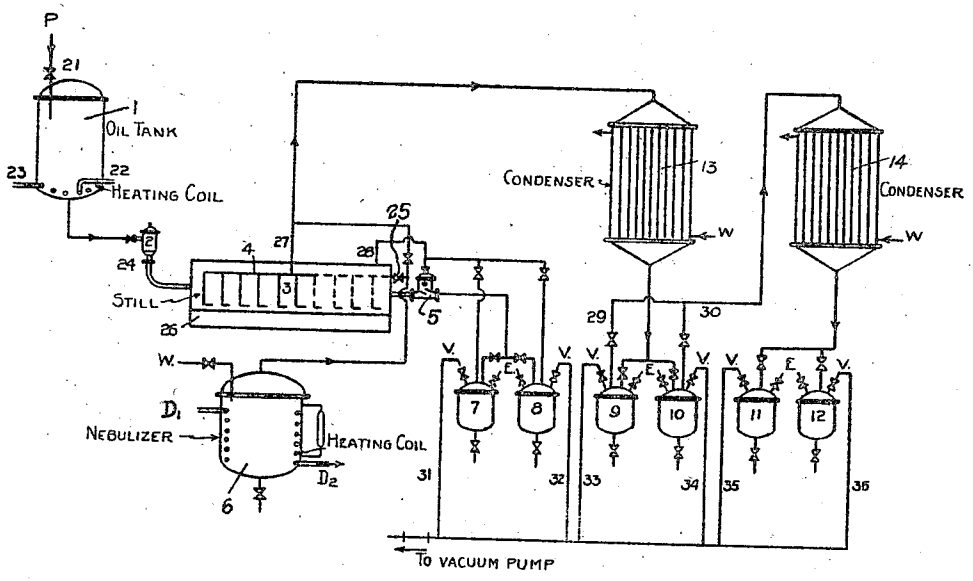

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RECOVERY OF OILS OF HIGH BOILING POINT

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It is known that when residues are distilled in order to recover oils of high boiling point, say more than about 300° centigrade, for example lubricating oils, considerable losses through decomposition easily occur, even when the distillation is conducted in vacuo or with the aid of steam.

We have now found that oils of the said kind may be recovered from liquid or solid residues obtained by the destructive hydrogenation of distillable carbonaceous materials, such as coals, tars, oils, and the like under pressure, with complete, or almost complete, avoidance of losses through decomposition, and therefore with an excellent yield, and with considerable saving of time, by introducing into the said substances, at temperatures exceeding 300° centigrade but lower than temperatures at which substantial cracking of the oils contained in said residues sets in. As a rule cracking begins at from 420° to 450° C., the particular cracking temperature is, however, dependent on the special kind of residue treated and at a pressure less than atmospheric, gases or vapors or mixtures of the same, these being equivalents for the purposes of the present invention and being hereinafter referred to for the sake of brevity as vapors, which vapors contain liquids of low boiling point, such as water, benzene, toluene, alcohol, benzine and the like in a liquid form, preferably in the state of mist, as for example, damp steam. The lower limit of pressures available depends on the nature of the liquid present in the said vapors. With wet steam, for example pressures above 13 millimetres mercury gauge must be employed since below this pressure no liquid water can exist at ordinary or higher temperatures. A current of gas or vapor containing mist may be produced in a simple manner for example by cooling a vapor, or a mixture of gas and vapor, to such an extent that a portion of the vapor condenses to a fine mist. The liquid may also be finely distributed in the gas or vapor in any other suitable manner, for example by atomization or like means. The initial materials for the process of the present invention are hereinafter referred to for the sake of brevity as "hydrocarbon residues".

The process according to the present invention is preferably carried out in an apparatus which enables the operation to be performed continuously. The current of vapor is preferably blown in transversely to the layer of material to be distilled, which for example is a horizontal shallow layer of distillation material, a few centimetres in depth, which moves or is moved continuously through a vessel, preferably a vacuum vessel, and maintained at a temperature above 300° centigrade. This method of working is hereinafter referred to as the cross-flow principle. The pressure employed may vary between wide limits, and in many instances it is preferably below 100 millimeters, mercury gauge.

The process according to the present invention may also be carried out simply by passing organic liquids such as benzine or liquid water into the aforesaid hydrocarbon residues which have a higher temperature than the boiling point of the liquid introduced.

The accompanying drawing shows diagrammatically a side elevation of a plant, which is very suitable for carrying out the process according to the present invention.

Numeral 1 denotes a reservoir for the oil to be distilled. Oil is passed into this reservoir by way of valve 21. The reservoir may be provided with a preheating device for example coils through which a heating medium is passed in at 22 and withdrawn at 23. From the preheater the oil passes by way of valve 24 into the vessel 2 which together with vessel 5 serves for regulating the height of the level in the still 3. The liquids of low boiling point in conjunction with which the gases are supplied to the oil to be distilled are nebulized in the nebulizer 6 provided with heating coils entering the nebulizer at D_1 and leaving it at D_2. The nebulized liquids are passed by way of valve 25 and device 4 to the oil to be distilled. The oil is heated by means of any heating medium supplied into the lower part 26 of the still. The vapors evolved in the distillation pass by way of pipe 27 and condenser 13 into the collecting vessels 9 and 10. The more volatile constitu-

ents not condensed in the condenser 13 pass from the said collecting vessels 9 and 10 by way of pipes 29 and 30 into the condenser 14 and are collected in vessels 11 and 12. The residues are collected in vessels 7 and 8. Each of the collecting vessels is connected by means of pipes 31, 32, 33, 34, 35 and 36 respectively with the vacuum pump.

The following examples will further illustrate the nature of the said invention which however is not limited thereto.

Example 1

A residue containing tarry and asphaltic matter obtained in the distillation of crude mineral oil, having a melting point of from 20 to 30 degrees centigrade is heated to about 350° centigrade and then treated with damp steam in a vacuum of 50 millimetres pressure. About 50 per cent of highly viscous oils is obtained and the residue is practically free from coke. By distillation in vacuo, or with superheated steam alone, smaller yields are obtained, the oils are less viscous, and the residues are of higher melting point and contain coke.

In a similar manner the most viscous lubricating oils may be distilled according to the process herein described without undergoing decomposition or having their properties impaired.

Example 2

A distillation residue containing asphaltic and tarry matter and mineral constituents of a destructive hydrogenation product of lignite is distilled, at from 370° to 395° centigrade and 80 millimetres pressure, with steam which, prior to admission into the still, is cooled in such a way that a substantial part of the steam has condensed to water in the form of mist. An oil of 15 Engler degrees (measured at 50° centigrade) viscosity distils over with great rapidity, without cracking occurring.

In continuous working, the said distillation residue is allowed to flow in a low layer, about 3 centimetres in depth, through a wide pipe, in which inlet tubes are arranged in succession to allow steam to be passed through on the cross-flow principle. The vapors are drawn off through a common main to the condenser, and the residue runs off continuously into a receiver.

What we claim is:—

1. A process for the recovery of oils of high boiling point from hydrocarbon residues resulting from the destructive hydrogenation of distillable carbonaceous materials which comprises introducing at a pressure less than atmospheric pressure vapors carrying finely distributed particles of liquids of low boiling point into a residue of the said nature and which is heated to a temperature above 300° C. but below the cracking temperature of the oils contained in the said residue.

2. A process for the recovery of oils of high boiling point from hydrocarbon residues resulting from the destructive hydrogenation of distillable carbonaceous materials which comprises introducing in a continuous manner of working and at a pressure below 100 millimetres mercury gauge vapors carrying finely distributed particles of liquids of low boiling point into a residue of the said nature and which is heated to a temperature above 300° C. but below the cracking temperature of the oils contained in the said residue.

3. A process for the recovery of oils of high boiling point from hydrocarbon residues resulting from the destructive hydrogenation of distillable carbonaceous materials which comprises introducing damp steam at a pressure less than atmospheric pressure but above 13 millimetres mercury gauge into a residue of the said nature and which is heated to a temperature above 300° C. but below the cracking temperature of the oils contained in the said residue.

4. A process for the recovery of oils of high boiling point from hydrocarbon residues resulting from the destructive hydrogenation of distillable carbonaceous materials which comprises introducing damp steam at a pressure below 100 millimetres but above 13 millimetres mercury gauge into a residue of the said nature and which is heated to a temperature above 300° C. but below the cracking temperature of the oils contained in the said residue.

5. A process for the recovery of oils of high boiling point from hydrocarbon residues resulting from the destructive hydrogenation of distillable carbonaceous materials which comprises introducing at a pressure less than atmospheric pressure vapors carrying finely distributed particles of liquids of low boiling point into a residue of the said nature and which is heated to a temperature above 300° C. but below 395° C.

6. A process for the recovery of oils of high boiling point from hydrocarbon residues resulting from the destructive hydrogenation of distillable carbonaceous materials which comprises introducing damp steam at a pressure less than atmospheric pressure but above 13 millimetres mercury gauge into a residue of the said nature and which is heated to a temperature above 300° C. but below 395° C.

In testimony whereof we have hereunto set our hands.

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