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2,203,645

PROCESS FOR THE DISTILLATION OF TAR OR PITCH

Filed Jan. 27, 1938

2 Sheets-Sheet 1

Fig.1.

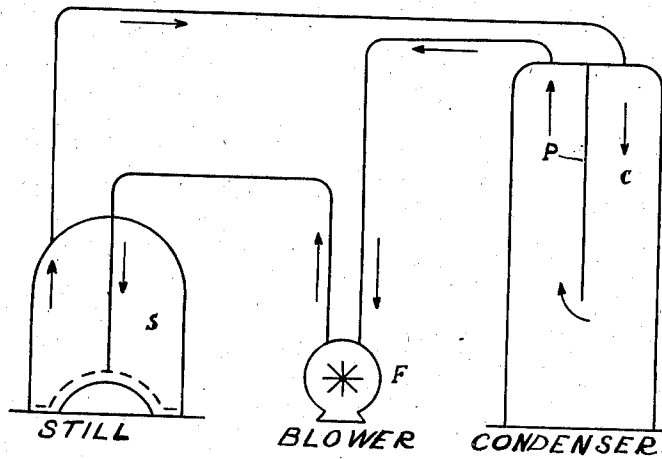
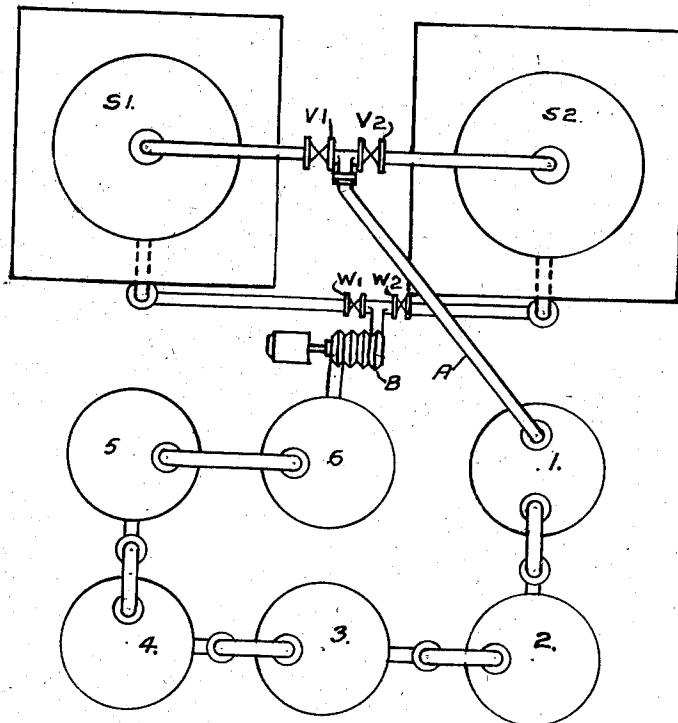


Fig.2.



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FIG. 3.

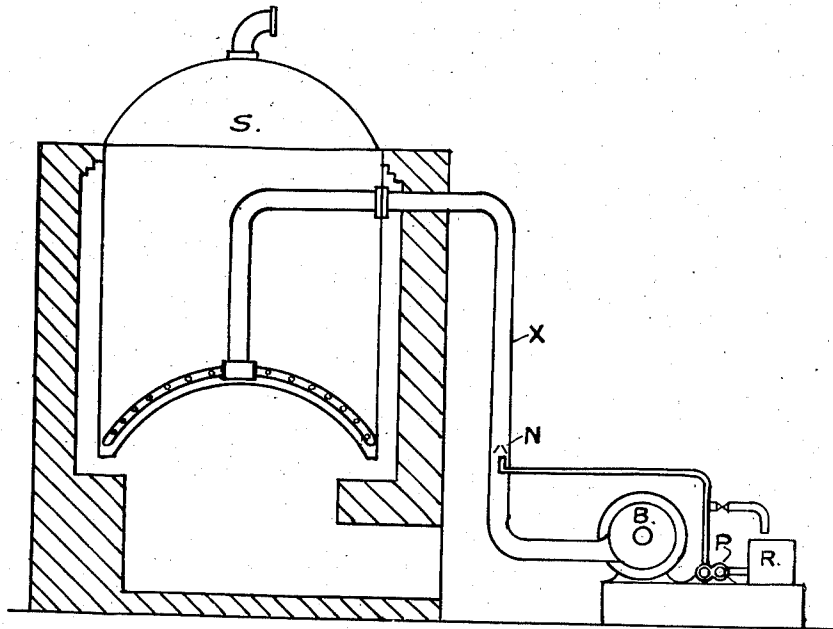
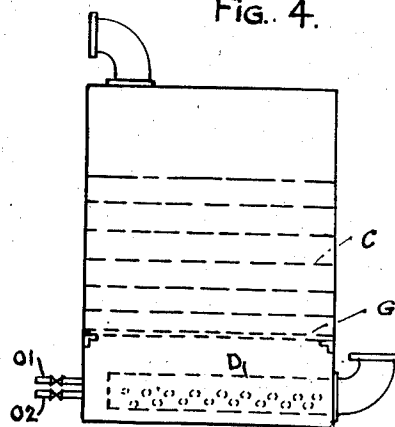


FIG. 4.



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PROCESS FOR THE DISTILLATION OF TAR
OR PITCH

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5 Claims. (Cl. 196—76)

It is known that if tar, derived from the carbonisation of coal, lignite, or other naturally occurring fuels, be distilled in a current of air, its separation into liquid distillates and residual pitch takes place more rapidly than by ordinary distillation. It is also known that such air blowing results in an increased yield of pitch with consequent decreased yield of distillates and that the residual pitch has a higher content of "free carbon" than ordinary pitch. It is known further that if a gas devoid of oxygen be used for blowing instead of air the distillation is accelerated in the same way, without increasing either the yield of pitch or its "free carbon" content.

The object of the present invention is to combine the acceleration of tar distillation with a considerably increased production of liquid distillates.

According to this invention, an oxygen-free gas, (which for the sake of brevity will be termed an "inert gas" in the following description), is circulated at a high velocity through an ordinary pot still in which tar is being distilled, into a relatively large air condenser or separator and thence back to the inlet of the totally enclosed fan, blower or compressor, which may be used to provide the circulation. A suitable inert gas may be nitrogen, carbon dioxide, coke-oven gas, coal gas, or the like, but it may be found convenient to use atmospheric air for one preliminary charge of tar, since the oxygen which it contains is rapidly absorbed by the heated tar, leaving at the end of a certain period of time nitrogen as the only gaseous constituent of the circulating medium.

After all the oxygen has been removed out of the circuit, the oxidised pitch present in the still is discharged and a fresh charge of tar is admitted into the still, for the purpose of the present invention proper.

The inert gas, in the course of its circulation, carries with it in the form of vapours and minute drops in suspension a relatively large amount of hydrocarbons and other volatile constituents of the tar which is being distilled. Owing to the sudden decrease in velocity of the gas, when it issues from the conduit which leads from the still to the separator, sedimentation as well as condensation of the distillates which accompany the gas, partially occur in the separator, at the bottom of which the distillates accumulate. It is, however, an all-essential feature of the present invention that such separation and/or condensation are only partial and that the inert gas returns to the circulating appliance heavily

charged, or supersaturated, with distillates in the form of vapour and drops in suspension, which may be conveniently alluded to as "fog." This feature distinguishes the invention from all processes in which efficient fractionation and/or condensation of the distillates take place before the propelling medium of gas re-enters the tar still.

There are two natural consequences of the method of procedure of the present invention. First, since there is no cooling of the inert gas (charged with vapours and fog) other than the cooling by radiation of the air condenser or separator, the temperature of the gas and vapours which return to the still rises rapidly with the progressive firing of the still. Secondly, since the whole system is enclosed and provision is made that the system be rigidly gastight and liquid-tight, an appreciable pressure develops inside the circuit. For instance, if coke-oven tar or gas tar be the raw material subjected to distillation, the maximum temperature to which the tar is raised may be in the neighbourhood of 300° to 330° C., when it will be found that the temperature of the returning gas, vapour and fog, is over 100° C. and the pressure inside the system has risen to a pressure equivalent to from 6 to 14 inches of a mercury manometer.

Although the treatment of tar by the above described process results in the production of an increased amount of liquid distillates for a residue of a desired consistency, as compared with ordinary distillation to a residue of the same consistency, appreciably greater increases are obtained as follows:

As soon as the material in the still has assumed the desired consistency, fresh tar is continuously pumped into the still, while the corresponding amount of pitch is withdrawn from it. In such a continuous mode of operation, the temperature of the still must be maintained constant at a figure which will generally be from 50 to 100 degrees centigrade below the boiling point of the highest-boiling constituent which it is desired to remove from the tar.

The conjoint action of a hot, inert gas, charged with tar-oil vapours and tar-oil fog, or tar maintained at the temperature indicated in the preceding paragraph, results in the production of from 35 to 50 per cent more liquid distillates than are produced by ordinary distillation to a residue of the same consistency, provided that the velocity of the gas and the temperature and internal pressure of the system are also main-

tained at the levels found by experience to be required.

The invention is equally applicable to tars and tar oils derived from high temperature and low temperature carbonisation, and works as readily with dehydrated tar as with tars which contain water.

An important feature of the invention relates to its application to tars containing water. Whereas it is found in all cases that the sum of the weights of residue and distillates produced by the invention exactly equals the weight of raw material treated, the weight of water associated with the distillates produced is less than the weight of the water present in the raw material.

By the method of the invention, therefore, a certain proportion of the water in the raw material combines chemically with one or more of the constituents of the raw material and, as a consequence of this chemical combination, the use of crude tars will in most cases be preferred to the use of dehydrated tars for the purpose of the present invention.

The most advantageous content of water in tar to be treated according to the present invention is from 4 to 6 per cent. Consequently when a tar is to be distilled which contains less than this amount of water, it is desirable to add sufficient water to the tar before treatment to bring the water content thereof up to from 4 to 5 per cent.

On the large scale a multi-stage fan, capable of circulating from 1000 to 2000 cubic feet of air per minute, may be conveniently used to distil coke-oven tar or gasworks tar in a pot still of 3000 to 4000 gallons' capacity at a rate of between 1½ to 3 tons of tar per hour. Owing to the high temperature of the circulating medium and to its content of hydrocarbon and phenolic constituents, both in the form of vapour and of fog, it is advisable to fit such a fan with gas and liquid-tight glands, thereby separating the interior of the fan from external, artificially cooled bearings.

The temperature and pressure of the circulating medium depend upon:

- (1) The temperature of distillation.
- (2) The rate of circulation.
- (3) The condensation and cooling effected by the air condenser.

Items (1) and (2) are fixed by the consistency of the pitch which is being manufactured and the rate at which it is desired to manufacture it. Item (3) depends on the capacity and exposed surface of the air condenser and the conduits leading from the outlet of the still back to the inlet of the fan, and is not greatly affected by atmospheric conditions in temperate climates, provided that the condensing system be protected from direct sunshine and rain. It is, however, best controlled by using a number of smaller vessels in series, instead of only one large vessel for the separation of the distillates.

The degree of supersaturation of the inert gas with tar-oil vapours and tar-oil fog, so far as hitherto described, is fortuitous depending on the rate of circulation of the gas and the temperature drop of the gas between the point where it leaves the heated tar and the point where it re-enters it.

An important feature of this invention is the means whereby the amount of supersaturation of the inert gas can be controlled and increased

to any extent desired, so that a further increase in yield of liquid distillates can be obtained.

In order to increase the supersaturation of the inert gas, the inert gas is passed from the still through a plurality of small condensers and/or fractionators in series and a desired quantity of condensate, consisting of the lighter constituents of the tar under treatment, or a mixture thereof with heavier distillates, is atomised and injected into the inert gas between the circulating device and the tar still, thereby increasing the supersaturation of the inert gas to any extent required, or is introduced directly into the liquid contents of the still at the discharge openings of the distributor for the inert gas in such a way that the spontaneous formation of a supersaturated mixture of the inert gas with atomised oil takes place inside the liquid contents of the still. The inert gas, charged in this way with a much greater proportion of accompanying tar-oil fog, sweeps out of the heated tar a correspondingly greater amount of liquid distillates.

It has been found that the lower boiling constituents of tar are most effective for this purpose and it is therefore advantageous to fractionate the distillates produced by the process as efficiently as possible. It has been found that very sharp fractionation results from the use of a large number of small condensing vessels in series, say six or more, of which one or more may contain columns of contact material of the desired form and height, or one or more may be in the form of bubblers. The object of the contact mass being to provide thorough contact between liquids and gases with the minimum resistance to the circulating flow, any of the well known forms of packing used in scrubbing appliances may be utilised, and thin rings of earthenware are found suitable for this purpose. Using six such vessels in series, numbered 1 to 6, No. 1 being nearest to the exit from the still and No. 6 being nearest the inlet of the circulating device, it will be found that, when coke oven tar is being distilled, vessels 1, 2 and 3 contain higher boiling fractions and vessels 4, 5 and 6 contain lower boiling constituents. The distillates containing light oils and/or light creosotes are most suitable for this purpose. In order to prevent excess of vapours and fog going to the circulating device, or excess of internal pressure in the system, either or both of the last two condensers and/or fractionators may be artificially cooled, for example by cold water or cold air.

According to the nature of the tar being treated, light oils from the requisite condensing vessels are run into a small container which may conveniently have a capacity of from 50 to 100 gallons. A suitable small pump of positive action and capable of exercising the necessary pressure, draws oil from this container and forces it through an atomising nozzle into the circulating stream at a suitable point in the conduit leading from the circulating device to the tar still, or the oil may be introduced directly into the liquid contents of the still. It may be found desirable to inject or introduce from 10 to 50 gallons or more of the specified light oil and/or creosotes into the circulating stream per hour.

Apparatus suitable for carrying out this invention is shown in the accompanying drawings, in which:

Fig. 1 shows an elevation, in diagrammatic

form of a plant using only one large condenser and one still;

Fig. 2 is a plan of a system comprising a pair of stills and six small condensers and/or fractionators in series;

Fig. 3 shows a sectional elevation of one still and blower and the appliance used for injecting atomised oil into the circulating system; and

Fig. 4 is an elevation on a larger scale of one fractionator.

As illustrated in Fig. 1, the inert gas passes from the still S into a large condenser C and back to the fan F. A vertical partition P may conveniently be fitted inside the condenser to prevent short-circuiting of the stream of gas and vapours in their passage back to the fan inlet.

As shown in Fig. 2, the two stills S1 and S2 are intended for use one at a time, so that each in turn can be cleaned out and prepared for charging, while the other is functioning for a desired period. A single conduit A connects either still with the fractionating plant, suitable valves V1 and V2 being provided to shut off one still while communication is made between the working still and the fractionators. In the same way, the return flow of the circulating medium from the blower B is directed by means of valves W1 and W2 into whichever still is working and is shut off from the still which is being rested.

In Fig. 3, S is the still through which the inert gas is directed by the blower B. A pump P draws light oil, or a suitable mixture of light and heavier oils, derived from the condensing system or fractionators 1 to 6, from a reservoir or container R and injects it through an atomising nozzle N into the pipe X leading from the blower B to the still S.

In Fig. 4, C is the contact mass supported on a grid G, D is the perforated pipe or other distributing appliance, O1 and O2 are overflow pipes fitted with cocks; the cock serving O2 is closed and the cock serving O1 is open for bubbling; the former is open and the latter is closed when bubbling is dispensed with.

It will be apparent that by suitably regulating the amount of light oil, or light oil and heavier distillates, injected into the stream of the inert gas, it is possible to regulate the amount of supersaturation of the inert gas to the desired extent and that therefore the supersaturation of the inert gas is under complete control, and can be varied at will.

Whereas it has been found that an inert gas, such as nitrogen constitutes the best vehicle for carrying the tar-oil vapours and tar-oil fog for the purpose of the invention, the process is not materially affected should the gas be wholly or partially replaced by steam and/or tar-oil vapours, so long as these become and remain supersaturated in the same way as is specified for the inert gas.

For instance, the plant may be purged with steam for the removal of air, and distillation started with the plant filled with steam at atmospheric pressure. As distillation proceeds, the enclosed steam will in due course become, first saturated and later, supersaturated with tar-oil vapours and tar-oil fog. Such procedure, however, is distinguished from other processes of steam distillation in that, once the plant has been filled with steam, no further addition of steam is required as continuous distillation proceeds. The water naturally present in, or purposely added to, crude tar will not only maintain all the steam which is required but will generally supply more

than is required, so that it may become necessary to condense the excess, together with some of the other vapours, before passing the circulating stream back to the circulating device. This condensation is easily effected, for instance as already described, by showering either or both of the last two vessels, 5 and 6, with jets of cold water and regulating the amount of cooling water so used to maintain the relative amounts of water vapour, tar-oil vapours and tar-oil fog so as to yield optimum conditions for the purpose of the invention.

A small flow of water suffices to give the necessary condensation and it has been found that from 100 to 300 gallons per hour of water at atmospheric temperature will maintain the desired composition of the circulating medium when 2 tons of crude coke oven tar are treated hourly.

It should be noted that this method of cooling the circulating medium for regulating its composition before its return to the circulating device is highly sensitive and capable of exact adjustment.

By this means, not only the desired proportions of vapours and fog are regulated, but also the internal pressure of the system is controlled, which might otherwise become excessive.

The present invention is furthermore applicable to any other system of tar distillation in the form of an additional unit. All that is necessary in such cases is to work the tar in the first system to an intermediate stage, such as refined tar or soft pitch, and to allow the residue of this first system to overflow continuously or periodically, by pumping or otherwise, into the additional unit which is in all respects similar to the plant hereinbefore described. This process can also be used if the still is charged with pitch, which has been treated by means of other processes, and a further quantity of liquid distillates can be obtained therefrom without altering the physical properties of the pitch as regards consistency and melting point.

I claim:

1. Process of distilling tar or pitch, which consists in circulating at a high velocity an inert gas, supersaturated with tar vapours, through the material being treated, said material being contained in a still, and through a plurality of condensers in series, atomising a desired quantity of condensate from the condensers and injecting it into the circulating medium at a point between the circulating device and the still, the condensers being maintained at such temperature that the amount of vapours condensed therein does not relieve the supersaturation of the circulating medium.

2. Process of distilling tar or pitch, which consists in circulating at a high velocity an inert gas, supersaturated with tar vapours, through the material being treated, said material being contained in a still, and through a plurality of condensers in series, and introducing a desired quantity of the condensate from the condensers directly into the liquid contents of the still at points suitable for effecting the spontaneous formation of a supersaturated mixture of gases, vapours and fog.

3. Process of distilling tar or pitch, which consists in circulating at a high velocity an inert gas, supersaturated with tar vapours, through a still containing the material being treated and a plurality of small condensers in series, at least one of which contains a column of contact material of the desired form and height, atomising a desired quantity of the condensate from the con-

densers and injecting it into the circulating medium at a point between the circulating device and the still.

4. Process of distilling tar or pitch, which consists in circulating at a high velocity an inert gas, supersaturated with tar vapours, through a still containing the material being treated and a plurality of small condensers in series, at least one of which is in the form of a bubbler, and introducing a desired quantity of the condensate from the condensers directly into the liquid contents of the still at points suitable for the effecting the spontaneous formation for a supersaturated mixture of gases, vapours and fog.

5. Process of distilling tar or pitch, which consists in circulating at a high velocity an inert gas, supersaturated with tar vapours, through a still containing the material being treated and a plurality of small condensers in series, at least one of which is in the form of a bubbler and contains a column of contact material of the desired form and height, and introducing a desired quantity of condensate from the condensers into the circulating stream, at a point suitable for the formation of a supersaturated mixture of gases, vapours and fog.

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