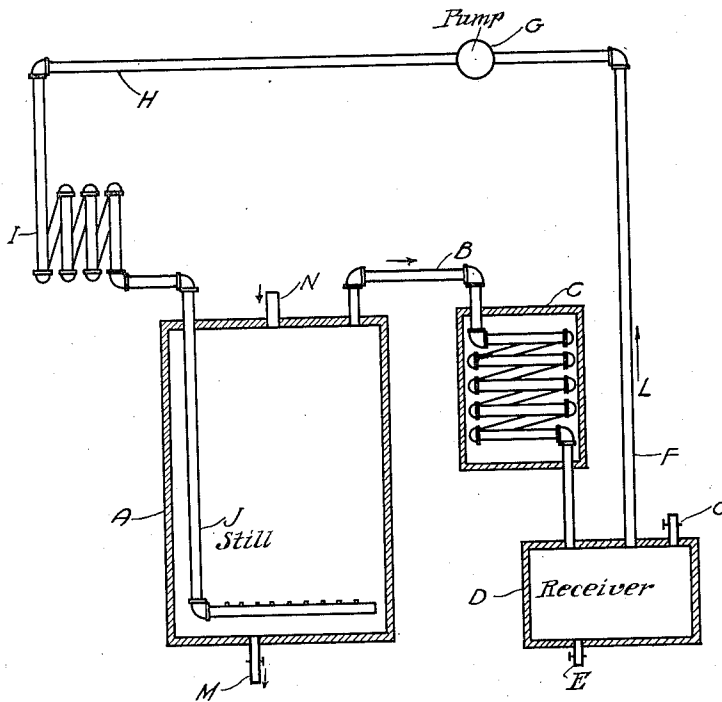


J. M. WEISS.
DISTILLATION PROCESS AND PRODUCT THEREOF.
APPLICATION FILED FEB. 7, 1920.

1,418,893.

Patented June 6, 1922.



INVENTOR

John M. Weiss

BY

Chas. W. Mortimer
ATTORNEY

UNITED STATES PATENT OFFICE.

JOHN M. WEISS, OF NEW YORK, N. Y., ASSIGNOR TO THE BARRETT COMPANY, A CORPORATION OF NEW JERSEY.

DISTILLATION PROCESS AND PRODUCT THEREOF.

1,418,893.

Specification of Letters Patent. Patented June 6, 1922.

Application filed February 7, 1920. Serial No. 356,849.

To all whom it may concern:

Be it known that I, JOHN M. WEISS, citizen of the United States, residing at New York city, in the county of New York and State of New York, have invented certain new and useful Improvements in Distillation Processes and Products Thereof, of which the following is a specification.

In the distillation of tars which are the products obtained from the destructive distillation of coal, lignite and similar organic substances, a progressive decomposition takes place as evidenced by the formation of a material high in carbon content and commonly known as "free carbon". The term "tar" as used in this specification is intended to include all varieties of coal tar, water, gas tar, oil gas tar, blast furnace tar, lignite tar, etc. In the distillation of these tars the greater the formation of "free carbon" the less the yield of distillate oil and the greater the residual pitch. As oil has in general a higher market value than pitch it is advantageous to obtain as high a yield of oil as possible. For this reason vacuum distillation has been used but applying this to the ordinary tar still involves constructional difficulties to safeguard the still against external pressure at high temperatures, and the vacuum necessary for the best results is difficult, if not impossible to maintain in practical operations. Moreover, when the tar temperatures reach a certain point, it is impossible to maintain a high vacuum, owing to uncondensable vapors given off by the decomposition of the tar unless a pump of such over capacity is used as to make the installation uneconomical and impractical.

Various gases have been proposed and used for agitating tar during the process of distillation, but the main purpose of this was to keep the carbon present in the tar or resulting from decomposition from forming on the shell of the still, thereby necessitating frequent cleaning in order to get suitable heat transfer, and no one has heretofore discovered that an increased oil production could be obtained by using particular gases for agitation, so far as I know. These gases heretofore used, such as steam, air, etc., have been used in comparatively small volume, just sufficient to maintain efficient agitation. I am not aware that anyone has, before my invention, used a suit-

able gas in sufficient amounts to obtain an appreciable increase in oil production from tar distillation over that which is obtained with straight air or steam distillation or that anyone else has discovered the effect of different gases in this respect.

I have discovered that if the right kind of gas is used in comparatively large amount to agitate the tar and is recirculated (in order to avoid loss of vapor mechanically carried by the gas and not liquified in the condenser) and more particularly if gases of certain specific chemical and physical properties are used that the yield of oil is materially increased over that of straight distillation or over that of distillation in which gases of different physical and chemical properties are circulated. This increased yield of oil is more pronounced the further the distillation is carried and in general reaches proportions of real practical importance when pitches are obtained which have melting points of approximately 170° F., or higher when tested by the cube in air method (Journ. Indus. and Eng. Chem. vol. 10, page 821), although quite marked differences have been noted in certain tars at much softer pitches and indeed at all stages of the distillation process. This application is a continuation, in part, of my application, Serial No. 303,950, filed June 13, 1919.

In my method the gas must be one which does not act chemically upon the tar. This eliminates gases containing substantial amounts of free oxygen as these gases act chemically on the tar. The gas must be a permanent gas at ordinary temperatures. Steam, for instance, is not suitable owing to the necessity of maintaining the entire system above 100° C. The gas must further be substantially insoluble in tar at the temperature of distillation.

Although I do not desire to limit myself strictly to the following theory, yet the results obtained seem best explained on the following theoretical basis.

Oxygen either alone or in form of air acts chemically on tar at higher temperatures and lowers the formation of oil during distillation. With some tars a mere passage of air during dehydration will result in an oxygen absorption sufficient to induce the oxygen content of the exit gases to less than five per cent and in a few cases the absorp-

tion of oxygen is almost quantitative. An inert insoluble gas will cause the oils to distil at a lower temperature owing either to the fact that it gives a mixed vapor pressure which follows the law of immiscible liquids or that the gas in some way acts on surface tension of the liquid so as to facilitate and speed the rate of saturation of the liquid particles in the gas and thereby accomplishing their volatilization with a lesser use of external heat.

The gas to be used should not act chemically upon the tar, it should be permanent at ordinary temperatures, and should be substantially insoluble in the tar. Of the more commonly known gases, nitrogen, carbon dioxide and carbon monoxide meet these requirements in a very excellent degree. I do not desire to limit myself to gases in a substantially pure state as it is obvious that a mixture of suitable gases in any proportions would be satisfactory. Further admixtures of small amounts of oxygen in the gases fall within the scope of the invention as it is obvious that the effects produced are probably dependent on the bulk constituent of the gas used and not on other substances present as impurities in small amount. It is further obvious that if air itself were used and recirculated, that in a very short time the oxygen would be chemically removed and substantially pure nitrogen would be left and from that point on the operation would be in accordance with my invention.

For best results an amount of gas not less than one cu. ft. (measured at ordinary temperatures and atmospheric pressure) per minute per 100 gallons of original material introduced in the still should be recirculated and an amount greater than 20 cu. ft. per minute per 100 gallons of original material to be distilled does not seem to present any substantial advantage in obtaining a higher yield of oil than the lesser amounts. It is also desirable to heat the circulated gas before introducing it into the still but this is not essential to the process.

As illustrative of how the process can be applied to tar distillation reference may be made to the appended diagrammatic illustration but it must be understood that I do not limit myself to any particular form of apparatus and the one shown is for illustrative purposes only.

A still A heated by any suitable means and provided with a drawoff M and a filling line N is connected by a pipe B to a condenser C which delivers into a receiver D with a drawoff E. From the top of the receiver a pipe F leads to a pump G and thence by a pipe H to a superheater I and by the pipe J to the bottom of the interior of the still communicating with it through the perforated pipe K. The inlet pipe O serves as a vent or for the introduction of gas into the

system. The arrows L show the direction of gas flow.

The still is charged with the tar to be distilled and the pump G started and a suitable gas is drawn in through pipe O until the system is filled. The system is then closed and heat applied, the gas being circulated at the desired rate. Distillation starts and is continued with the circulation maintained until the residue in the still is of the desired consistency, the volatile constituents being carried off by the circulating gas from still A and condensed in the condenser C. The heat and gas circulation are then discontinued and the residue is removed from the still A.

It is obvious that the process is independent of the size or type of still used, the type of setting, the method of heating, the type of or means of cooling the condenser, etc. The important factor is that the gas used shall have the proper physical and chemical characteristics.

The following results illustrate the advantage which may be obtained by the use of this process in the distillation of a by-product coke oven tar. The particular tar used had a free carbon content of 8.5%. Passage of gas in all cases was made at the rate of about 15 cu. ft. per minute per 100 gallons of material introduced in the still.

	Per cent by weight oil obtained.	Per cent by weight pitch obtained.	Pitch air melting point.	Pitch per cent free carbon.
Distillation without agitation.....	40.1	57.8	280° F.	40.7
Air used not recirculated.....	33.0	63.0	286° F.	35.1
Nitrogen recirculated.....	52.0	46.4	277° F.	27.2
Carbon dioxide recirculated.....	51.2	46.7	270° F.	28.2
Mechanically stirred.....	40.0	57.9	300° F.	46.5

The advantages of the applicant's process are very evident. Air instead of increasing the yield of oil has decreased it due to oxidation while the preferred gases have materially increased the oil yield over that obtained by the other methods.

A further advantage is obvious from the fact that in the ordinary distillation of a tar to a hard pitch it is advisable to stop the distillation at a pitch of about 280° F. melting point for above this point there is a very decided tendency to form coke upon the shell of the still with the consequent danger of blistering or buckling the same due to the poor heat transfer to the body of the charge. Such action is indicated by the data given above in this application. On account of the relatively small carbon formation resulting from my process, the distillation may be carried to such an extent that a pitch of a much higher melting point may be produced without endangering the apparatus, and consequently a much further greater yield of oil may be obtained than by methods already known to the art. By my process there is

less of the oil decomposed and the oil can be more completely removed from the pitch than was the case with prior processes.

Moreover, to the best of my knowledge, the highest melting point of pitch obtained in the past in practical plant operations has not been over 320° F. determined as referred to above. There are statements in the literature that it is possible to obtain coal tar pitches with fusing points as high as 345° F. but the further statement is made that these are rarely if ever met with in practice, indicating that such pitches if made were only the result of laboratory experiments. By my method I have readily succeeded in obtaining pitches of a melting point of 350° F. to 600° F. and these have been capable of being removed from the stills without difficulty or undue coking on the shell of the still. Such pitches can be readily powdered for subsequent use and will not cake together even when exposed to the highest temperatures due to weather conditions. In commercial operations pitch is often afterward coked in various forms of apparatus. With ordinary pitches as usually produced, a considerable foaming takes place during the coking operation so that the vessels used can only be filled to a comparatively small fraction of their capacity. With the harder pitches from the new process much less foaming takes place during coking so that a much larger proportion of the vessel used for coking may be utilized, resulting in a much greater capacity for a given installation.

I claim:

1. A process of tar distillation comprising the repeated circulation of a gas through hot tar at a rate of from 1 to 20 cu. ft. per 100 gals. of tar, per minute, said gas being uncondensable at 0° C. and atmospheric pressure, not exerting chemical action on the tar and relatively insoluble in the tar, substantially as described.

2. A process of tar distillation comprising the repeated circulation of a mixture of gases through hot tar at a rate of from 1 to 20 cu. ft. per 100 gals. of tar, per minute, said

gases being uncondensable at 0° C. and atmospheric pressure, not exerting chemical action on the tar and relatively insoluble in the tar, substantially as described.

3. A process of tar distillation comprising the repeated circulation of nitrogen through hot tar at a rate of from 1 to 20 cu. ft. per 100 gals. of tar per minute substantially as described.

4. A process of tar distillation comprising the repeated circulation of gas through a hot tar, said gas being uncondensable at 0° C. and atmospheric pressure, not exerting chemical action on the tar and relatively insoluble in the tar, and continuing said recirculation until the residuum has a melting point above 350° F.

5. A process of tar distillation comprising the repeated circulation of nitrogen through hot tar until the residuum has a melting point above 350° F.

6. A process of tar distillation comprising the repeated circulation of a gas through hot tar at a rate per minute of from 1 to 20 cu. ft. per 100 gals. of tar, said gas being uncondensable at 0° C. and atmospheric pressure, not exerting chemical action on the tar and relatively insoluble in the tar, and continuing said recirculation until the residuum has a melting point above 350° F.

7. A process of tar distillation comprising the repeated circulation of nitrogen through hot tar at a rate per minute of from 1 to 20 cu. ft. per 100 gals. of tar, until the residuum has a melting point above 350° F.

8. As an article of manufacture, a pitch obtained by the distillation of tar, said pitch having a melting point about 350° F.

9. As an article of manufacture, a pitch obtained by the distillation of tar, said pitch having a melting point about 430° F.

10. As an article of manufacture, a pitch obtained by the distillation of tar, said pitch having a melting point of at least 430° F. and being substantially non-foaming when destructively distilled to coke.

In testimony whereof I affix my signature.

JOHN M. WEISS.

CERTIFICATE OF CORRECTION.**Patent No. 1,418,893.****Granted June 6, 1922, to****JOHN M. WEISS.**

It is hereby certified that error appears in the printed specification of the above numbered patent requiring correction as follows: Page 3, line 86, claim 8, for the word "about" read above; and that the said Letters Patent should be read with this correction therein that the same may conform to the record of the case in the Patent Office.

Signed and sealed this 7th day of April, A. D. 1931.

(Seal)

M. J. Moore,
Acting Commissioner of Patents.