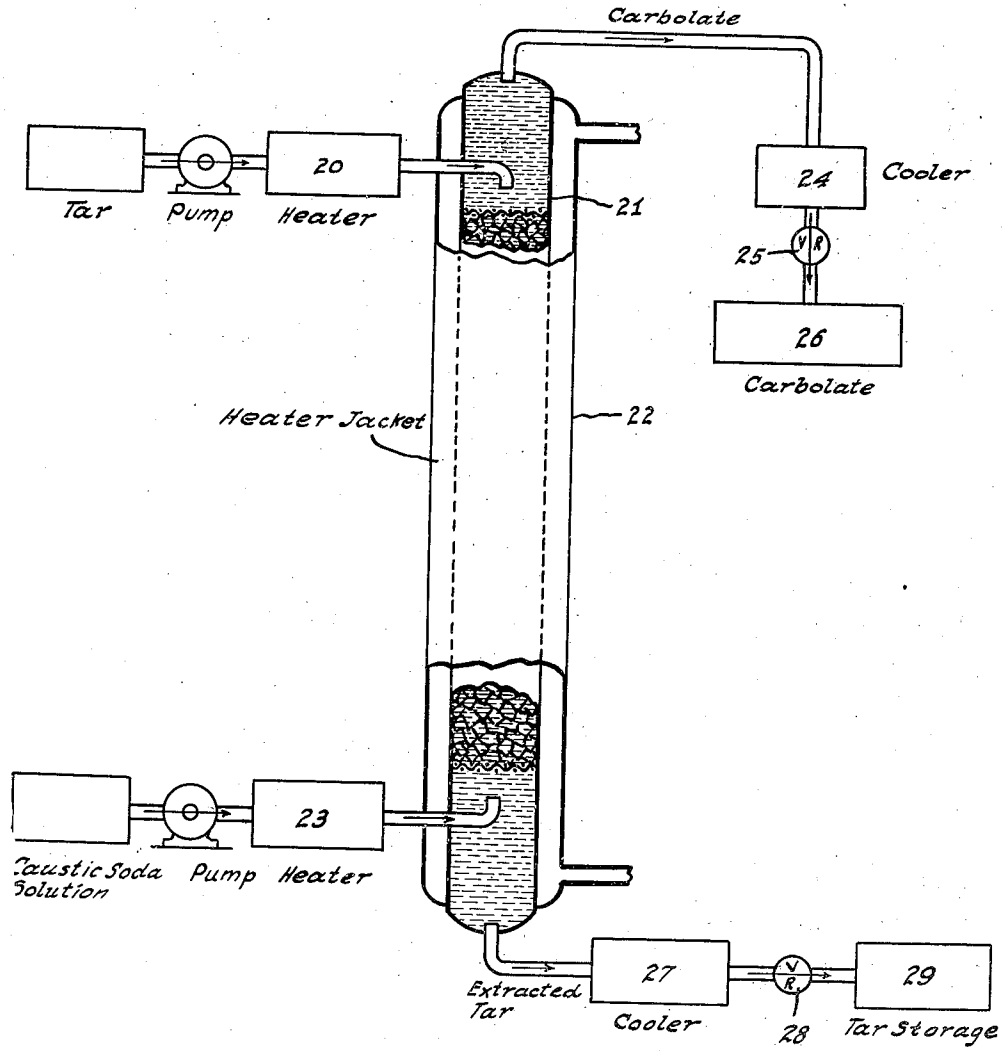
March 11, 1947. F. E. CISLAK ET AL 2,417,291

PROCESS OF SEPARATING TAR ACIDS FROM TAR

Filed July 19, 1943



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Patented Mar. 11, 1947

2,417,291

UNITED STATES PATENT OFFICE

2,417,291

PROCESS OF SEPARATING TAR ACIDS FROM TAR

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Application July 19, 1943, Serial No. 495,314

5 Claims. (Cl. 260—627)

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This invention relates to an improved process for separating tar acids from coal-tar. More specifically it relates to a simple process of removing tar acids directly from the coal-tar without producing large amounts of undesired by-products.

Coal-tar as ordinarily incidentally produced in the manufacture of coke or coke-oven gas contains tar acids in amounts varying from about ½% to 8%. These tar acids, especially phenol, cresols, and xylenols, are urgently needed in our war effort, and it is most important that a simple and efficient means be used to recover them from the coal-tar.

In order to recover the tar acids from the tar it has heretofore been necessary first to subject the tar to distillation, and then to extract the tar acids from the distillate oil. The extraction of the tar acids from the distillate oil ordinarily consists in intermixing that oil with an excess of aqueous caustic soda solution, thereby converting the tar acids into their sodium salts. These sodium salts, commonly referred to as carbolates, are soluble in the aqueous caustic soda solution; and are subsequently separated from the residual oil, which is insoluble in the aqueous caustic soda solution, by allowing the mixture to stand to produce an aqueous layer and an oil layer and then separating the two layers, as by decanting. The tar acids have then been recovered from the aqueous layer, in known manner.

This heretofore customary method of recovering tar acids from coal-tar is uneconomical, in that it requires the distillation of the tar merely to recover the ½% to 8% of tar acids present therein, and such a distillation produces as by-products large amounts of oil and pitch for which there is relatively little demand. Because of this lack of economy, the recovery of tar acids has often not been done; and a great deal of coal-tar has been burned as fuel, or sold for road-building purposes, without recovering the greatly needed tar acids.

Many unsuccessful attempts have been made to extract the tar acids directly from coal-tar, by the use of aqueous caustic soda solutions. However, when an aqueous caustic soda solution is intermixed with coal-tar at ordinary temperatures, or even at elevated temperatures under 100° C., instead of subsequently separating into the desired aqueous layer and tar layer an undesirable rather stable emulsion is obtained. Such an emulsion is undesirable because it is extremely difficult, if not impossible, to separate a tar-free aqueous caustic soda solution of tar acids from it; and also it is extremely difficult, if not impossible, to separate a caustic-free tar from it. It is important that a clean separation of layers be obtained; for the tar acids cannot be economically purified unless the caustic soda solution is reasonably free of tarry material, and the tar is unsuitable for use, for instance as a fuel, if it contains more than a trace of caustic soda.

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We have now found that if coal tar and aqueous caustic alkali (caustic soda or caustic potash) solution are intermixed at a temperature substantially above 100° C. (preferably below 300° C. and desirably within the range of 125° C. to 175° C.) and at a pressure above the vapor pressure of the mixture at this temperature, and then allowed to separate into layers while maintaining both temperature and pressure, and then the separated layers are cooled to a temperature below 100° C. and the pressure is reduced to atmospheric pressure either simultaneously with or before or after the cooling, we avoid emulsion difficulties, and obtain a substantially tar-free aqueous caustic-alkali layer containing the tar acids, and a substantially caustic-free tar layer.

The temperature at which the coal-tar and the caustic soda solution are intermixed must be sufficiently above 100° C. so that the viscosity of the tar is low enough to allow the ready separation of the aqueous and tar phases. This temperature will vary somewhat with different tars, but in general will fall within the range of 125° C. to 175° C.

The pressure at which the tar and caustic soda solution are intermixed and allowed to separate into layers must be equal to or greater than the vapor pressure of the mixture at the temperature used, so that there will be no boiling; and is desirably at least 10 pounds per square inch above that vapor pressure. Pressures of the order of 50 to 150 pounds per square inch are desirable when the temperature used is between 125° and 175° C.

The single figure of the accompanying drawing shows diagrammatically an illustrative apparatus suitable for carrying out our invention.

In that illustrative apparatus, coal tar is pumped continuously through a heater **20**, where it is heated to the previously determined suitable operating temperature, and then is introduced under pressure near the top of a counter-current extraction column **21**; which is also heated to said operating temperature, as by a heating jacket **22**, and in which the predetermined operating pressure is suitably maintained, as by the pumps shown. Aqueous caustic soda solution is pumped

continuously through a heater 23, where it is heated to the predetermined operating temperature, and then is introduced under pressure near the bottom of the extraction column 21. The caustic soda solution rises through the descending tar, in the extraction column; and as it does so it extracts the tar acids therefrom by converting them to water-soluble carbolates, and carries those carbolates to the top of the column 21. The solution of carbolates is continuously removed from the top of the column 21, and is passed through a cooler 24 and then through a pressure-reducing valve 25 to a carbolate receiver 26, in which latter the pressure is reduced to about atmospheric pressure. The extracted tar is removed continuously from the bottom of the column 21, and is passed through a tar cooler 27 and then through a pressure reducing valve 28 to a tar receiver 29, in which latter the pressure is reduced to about atmospheric pressure. The tar acids are recovered from the carbolate solution in known manner.

The aqueous caustic soda must have a specific gravity different from that of the coal tar. We prefer to use aqueous caustic soda of a concentration such that its specific gravity, at the operating temperature, is less than that of the coal tar; although we may use one which has a specific gravity greater than that of the coal tar. For most tars we find an aqueous caustic soda of about 3% to 6% to be satisfactory. We also prefer to use an amount of caustic soda considerably in excess of that required to react with all the tar acids present in the tar, thereby facilitating the removal of all the tar acids.

While we prefer to practice our invention in the continuous manner described, we do not limit our invention to such a continuous process; for our invention is also applicable to batch operation. In practicing our invention by the batch method, tar and caustic soda are introduced, with little and desirably no preliminary mixing, into the extraction vessel, the vessel is closed, and the contents are heated to the previously determined operating temperature, conveniently about 125°–175° C.; then the contents are thoroughly intermixed, after which they are allowed to stand to separate into two layers. then the whole is cooled to about 90° C. and the pressure is released, and then the two layers are withdrawn separately.

In either the continuous process, as carried out for example in the apparatus shown in the drawing, or in the batch process, the caustic soda layer obtained is substantially free from tar; and the tar layer obtained is sufficiently free from caustic soda for most purposes. However, if an extremely caustic-free tar is desired, it may be obtained by treating the separated tar layer as follows:

One part of the tar layer is mixed with 0.15 part to 0.30 part of water, and thoroughly agitated at 80° to 90° C. The resulting mixture of water and tar is allowed to separate into two layers while being maintained at the temperature of 80° to 90° C. This usually requires from 1 to 6 hours. The separated tar layer is now extremely caustic-free, as the caustic has been extracted by the water from the tar.

We claim as our invention:

1. The process of separating tar acids from coal tar, which consists in mixing the coal tar containing the tar acids with an aqueous caustic alkali solution at a temperature substantially above 100° C. and at a pressure above the vapor pressure of the mixture at the temperature used, allowing the mixture to separate into a tar-acid-containing aqueous layer and a tar layer while maintaining both temperature and pressure above the aforesaid values, and withdrawing one layer.

2. The process of separating tar acids from coal tar, which consists in mixing the coal tar containing the tar acids with an aqueous caustic alkali solution at a temperature substantially above 100° C. and at a pressure above the vapor pressure of the mixture at the temperature used, allowing the mixture to separate into a tar-acid-containing aqueous layer and a tar layer while maintaining both temperature and pressure above the aforesaid values, and withdrawing the two layers separately.

3. The process of separating tar acids from coal tar as set forth in claim 1, in which the pressure used is at least 10 pounds above said vapor pressure.

4. The process of separating tar acids from coal tar as set forth in claim 1, in which the temperature is between 125° C. and 175° C., and the pressure is between 50 pounds and 150 pounds per square inch.

5. The process of separating tar acids from coal tar as set forth in claim 1, in which the cooling of the separated layers is done while maintaining the pressure.

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