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PROCESS FOR RECOVERING PHENOLS FROM PHENOL-CARRYING DISTILLATION GAS AND THE LIKE

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The injurious effects produced by the phenols which find their way into the effluent of coke oven plants, gas works and carbonizing plants, make it necessary to treat the effluent for removal of these bodies.

It has been attempted to purify the effluent from the ammonia recovery plant but it has been found to be more advantageous to apply the purification to the ammoniacal liquor before this has been treated for the recovery of ammonia.

The basis of the process is the idea that phenol is most easily recovered when it is at as high a concentration as possible. Since the hot gases from the coke ovens contain the phenol in a concentration of about 2 grams per cubic meter, while the concentration in the ammoniacal liquor separated from the gas is of the order of about 4000 grams per cubic meter, and since the liquor is further diluted by subsequent operations, it could not be expected that it would be possible to recover the phenol more easily and more cheaply at any other point in its path from the oven to the drains than from the crude ammoniacal liquor.

However, the unexpected is the case for it has been found that according to the present invention it is possible in a simple manner to wash completely the phenol from the gases more easily and more economically than from the ammoniacal liquor in which the phenol has a concentration 2000 times as great.

For this purpose the gas which is in the neighborhood of its dew point and still contains substantially the whole of its water in the form of vapor is brought, preferably after it has been freed from its content of tar, into contact, preferably in counter-current, with a wash liquor suitable for absorbing the phenols, for instance benzol wash oil, heavy benzol, another coal tar oil or brown coal tar oil, or a liquid of similar action. The gas parts with its phenols to the wash liquor and in the subsequent cooling of the gas there is produced a liquor free from phenols. The wash liquor charged with phenols may be recovered in known manner, for example by washing it with caustic soda lye or by distillation and may be used again.

The advantage of the process of the invention for washing phenols from gas as compared with the usual method of removing it from ammoniacal liquor is that the wash oil can be enriched to a higher percentage with phenols by contact with the gases than by contact with water in the liquid state. In the latter case it has been found in practice that in rational working the phenol content of the wash liquor may be increased to about double the content of phenol in the ammoniacal liquor.

On the other hand, by operating according to this invention the phenol content of the wash oil may be some 10 times that which the ammoniacal liquor has. In order to remove a given quantity of phenol, therefore, there need be used only one-fifth of the quantity of wash liquor which is necessary according to the former procedure. Moreover, the speed of the absorption of the phenol from the gas into the wash oil is incomparably greater than the absorption into the wash oil from the ammoniacal liquor. Whereas in the former process the wash oil is in contact with the ammoniacal liquor for some hours, the process of this invention only requires a few seconds for the absorption of the phenol by the wash oil.

Since both the quantity of wash oil to be used and the duration of the operation constitute only a fraction of the quantity and time necessary in the earlier processes, it is apparent that the apparatus concerned may be considerably smaller than in former installations. Since at the same time the quantity of liquid to be moved is smaller the consumption of power is correspondingly smaller.

A further great advantage of the new process resides in the fact that it is easier to recover the phenol from the wash oil which has been used in the process because its concentration is higher. According to the earlier processes about 20 cubic meters of oil on the average had to be worked up for the recovery of 100 kilos of phenol, whereas by the present invention only about 4 cubic meters of oil must be so worked up.

It is obvious that the problem in the latter case is more cheaply solved irrespective of whether the phenol is to be recovered by distillation or by extraction.

A further very substantial advantage in working according to this invention resides in the following fact:—In washing ammoniacal liquor with a solvent the several components of the mixture of phenols pass into the oil in accordance with their distribution ratio in respect of the two liquids, which is dependent on their solubility in water. The carbolic acid is by far the most freely soluble. It is therefore washed out with the greatest difficulty. In all crude phenols or phenolate lyes which are produced in the recovery of phenols from ammoniacal liquor, the carbolic acid is contained in considerable smaller proportion than in the ammoniacal liquor from which the products have been recovered. In washing the gas, on the other hand, the components of

the phenol mixture pass into the wash oil correspondingly with their vapor pressure. Since this in the case of the phenols concerned in practice is substantially the same, the product obtained by removing the phenol from the gases contains the several components and therefore the carbolic acid also in about the same proportions as they exist in the ammoniacal liquor. Since, however, the carbolic acid is the most valuable part of the product recovered the price of this product recovered by use of this invention is considerably higher on account of its higher percentage of phenol than that recovered in the usual processes.

Another advantage resides in the following fact:—As is known, the ammoniacal liquor contains substances of more strongly acid character than that of the phenols. The solubility of these substances in water is so great that when the ammoniacal liquor is washed with a solvent they are separated only in small degree. They remain as impurities in the effluent. On the other hand, the vapor pressure of these substances is very small; therefore in working by the present invention they are completely removed.

For illustrating the essence of the new process there may be cited as an example removal and recovery of the phenols of a coke oven plant having a working capacity of 1000 tons of coal in 24 hours. The gas, which consists of a mixture of gases and phenols is preferably freed from tar and maintained at a temperature above its dew point (for example 80° C.) and containing more than 40 grams of water per cubic meter, enters at the bottom of a tower of 3 meters diameter and 6 meters high, which is half filled with bodies of the nature of Raschig-rings. In the gaseous mixture being treated, at the temperature indicated, the dewpoint differs from that of the air, and water content has been ascertained to be over 40 grams per cubic meter of gas mixture. Into the upper part of the tower is delivered through nozzles, sprays, distributing pipes or the like 1 cubic meter per hour of benzol wash oil. The wash oil which leaves the tower contains about 30 grams per litre of phenols. From the gases leaving the upper part of the tower, the ammoniacal liquor is separated on cooling which, according to local conditions, has a content of phenol varying from 0.05–0.2 grams per litre. The proportion of phenols withdrawn from the gaseous mixture amounts to about 90 per cent. of that present.

The wash oil leaving the tower is washed in known manner with concentrated caustic soda lye, preferably in two stages. Should it be desired to recover the bases, it is only necessary to treat the wash oil further, for instance with sulphuric acid.

What I claim is:—

1. The process for recovering phenols from phenol bearing distillation gas consisting of a mixture of gases with volatilized phenols together with an amount of water vapor exceeding 40 grams of water per cubic meter of the distillation gas, which process consists in first causing a suitable organic solvent to act upon said distillation gas in continuous counter-current at a temperature above the dewpoint of said gas, and subsequently recovering the phenols from said solvent.

2. The process for recovering phenols from a phenol bearing distillation gas mixture consisting of a mixture of gases with volatilized phenols in the presence of water vapor, which process consists in first causing a suitable organic solvent to act upon said gas mixture in continuous counter-current while at a temperature above the dewpoint of the gas mixture, and subsequently recovering the phenols from said solvent by extracting the same therefrom by means of an alkaline reagent.

3. The process for recovering phenols from a phenol bearing distillation gas mixture consisting of a mixture of gases with volatilized phenols together with an amount of water vapor to the extent of at least 40 grams of water per cubic meter of gas mixture, which process consists in causing an organic solvent to act upon said gas mixture in continuous counter-current while at a temperature above the dewpoint of the gas mixture, and subsequently recovering the phenols from said solvent by distillation.

4. The process for recovering phenols from phenol bearing distillation gas consisting of a mixture of gases with volatilized phenols together with an amount of water vapor exceeding 40 grams of water per cubic meter of the distillation gas, which process consists in first causing a mineral oil to act upon said distillation gas in continuous counter-current at a temperature above the dewpoint of said gas, and subsequently recovering the phenols from the said oil.

5. The process for recovering phenols from a phenol bearing distillation gas mixture consisting of a mixture of gases with volatilized phenols in the presence of water vapor, which process consists in first causing a suitable mineral oil to act upon said gas mixture in continuous counter-current while at a temperature above the dewpoint of the gas mixture, and subsequently recovering the phenols from the said oil by extracting the same therefrom by means of an alkaline reagent.

6. The process for recovering phenols from a phenol bearing distillation gas mixture consisting of a mixture of gases with volatilized phenols together with an amount of water vapor to the extent of at least 40 grams of water per cubic meter of gas mixture, which process consists in causing a mineral oil to act upon said gas mixture in continuous counter-current while at a temperature above the dewpoint of the gas mixture, and subsequently recovering the phenols from the said oil by distillation.

7. The process of recovering phenols from phenol bearing distillation gas including a mixture of gases and volatilized phenols together with water vapor, which process consists in treating said mixture in the presence of the water vapor with a mineral oil while the water content remains in the gas mixture, and subsequently recovering the phenol from said mineral oil.

8. The process for recovering phenols from phenol bearing distillation gas consisting of a mixture of gases with volatilized phenols together with an amount of water vapor exceeding 40 grams of water per cubic meter of the distillation gas, which process consists in first causing tar oil to act upon said distillation gas in continuous counter-current at a temperature above the dewpoint of said gas, and subsequently recovering the phenols from the said oil.

9. The process for recovering phenols from a phenol bearing distillation gas mixture consisting of a mixture of gases with volatilized phenols in the presence of water vapor, which process consists in first causing a suitable tar oil to act upon said gas mixture in continuous counter-current while at a temperature above the dewpoint of the gas mixture, and subsequently recovering the

phenols from said oil by extracting the same therefrom by means of an alkaline reagent.

10. The process for recovering phenols from a phenol bearing distillation gas mixture consisting of a mixture of gases with volatilized phenols together with an amount of water vapor to the extent of at least 40 grams of water per cubic meter of gas mixture, which process consists in causing tar oil to act upon said gas mixture in continuous counter-current while at a temperature above the dewpoint of the gas mixture, and subsequently recovering the phenols from the said oil by distillation.

11. The process of recovering phenols from phenol bearing distillation gas including a mixture of gases and volatilized phenols together with water vapor, which process consists in treating said mixture in the presence of the water vapor with tar oil while the water content remains in the gas mixture, and subsequently recovering the phenol from said oil of the tar oil type.

12. The process of recovering phenols from phenol bearing distillation gas including a mixture of gases and volatilized phenols together with water vapor, which process consists in treating said mixture in the presence of the water vapor with a solvent oil while the water content remains in the gas mixture, and subsequently recovering the phenol from said solvent oil.

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