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TREATMENT OF TARS

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This invention relates more particularly to the treatment of crude tars such as those obtained in the distillation of various naturally occurring solid carbonaceous materials including coals, lignites and shales.

In a more specific sense the invention has reference to an improved process for the extraction and recovery of the "tar acids" contained in various crude tars, and distillates or their fractions. The crude tars which may be treated by the process of the present invention vary considerably in character according to the source of the materials distilled, the conditions of distillation such as the temperature and pressure employed and the use of steam or inert gases as carrying agents. The quality of the tars is influenced to a great extent by the temperature employed. For example, the tars produced from coal at temperatures above 1400° F., and known to the trade as high temperature tars are generally characterized by lower viscosity, higher aromatic hydrocarbon content and lower phenolic content than tars produced at lower temperatures and generally classified as low temperature tars. In a general way the differentiation on a basis of temperature between high temperature and low temperature coal tars may also be applied to tars from lignite, peat, shale, etc., although the range of temperatures corresponding to high and low temperature tars will vary to some extent with the character of the material distilled.

Tars of the character under discussion consist for the most part of hydrocarbons of both a straight chain and a cyclic character, the proportions varying with the temperature of distillation, and also contain varying quantities of oxygen, nitrogen and sulphur compounds. The oxygen compounds include various compounds of a phenolic and naphtholic character, the recovery of which constitutes the subject of the present invention. The nitrogen compounds include amines and heterocyclic compounds such as pyridine, picolines, lutidines, etc., as well as some pyrrolle derivatives. The sulphur compounds include principally thiophene and its derivatives and homologs.

The phenolic constituents of tars which frequently comprise substantial percentages thereof are extremely useful as disinfectants and preservatives and as intermediates for the manufacture of more complicated chemical derivatives such as dyes, explosives, etc., and hence possess a particular commercial value. These compounds are of a weakly acid character (hence the name "tar acids") and react with varying intensity with different alkaline reagents to form compounds which may be generally grouped under the broad term of phenolates. When treating tars of varying quality with alkali solutions for the extraction of tar acids, difficulty is frequently encountered in the formation of extremely persistent emulsions owing possibly to the soap-like character of the phenolates and the presence of protective colloids. The formation of such emulsions slows up the treating step and decreases the capacity of equipment to a large extent.

In one specific embodiment the invention comprises treatment of crude tars containing acidic compounds with aqueous caustic soda of approximately 25° Baumé strength at temperatures from 140 to 160° F.

I have established as the result of a considerable number of experiments that the use of the above mentioned reagent and conditions of treatment correspond to practically complete absence of emulsions when treating crude tars for the extraction and recovery of their tar acid content. 25° Baumé caustic soda corresponds to a percentage of approximately 18.5 NaOH by weight, and it appears that this particular concentration when used in an amount corresponding to the neutralization of the acid content of the tar and within the temperature range indicated constitutes a solution of a critical concentration in respect to ease and efficiency of operation. The preferred temperature range indicated above insures a sufficient lowering of the viscosity of the tars without unduly increasing the vapor pressure of any volatile constituents which may be present. However, in the case of tars containing low boiling components, pressure may be employed to prevent ebullition and the consequent tendency toward froth production.

The process of the invention may be conducted in either batch or continuous apparatus after a titration of a sample of the tar has shown the approximate amount of alkali necessary for combination with the acidic constituents. As a rule, the most satisfactory results are obtained in the present instance when somewhat more than the equivalent amount of alkali is employed in the extraction, though the necessary excess is not large. In batch operations vigorous agitation by means of paddles or propellers may be employed to insure effective contact of alkali and tar without danger of emulsion formation. Treatments may also be conducted in continuous treating equipment consisting of alternate mixing and settling devices, the preheated tar and caustic

solution being pumped into the system at a point preceding a pipe containing baffling material and the solution of phenolates separated from the remaining tar compounds in a subsequent settler.

I claim as my invention:

1. A process for the treatment of tars containing tar acids to remove the said tar acids and prevent emulsification thereof, which comprises subjecting the said tars to the action of an aqueous solution of an alkali comprising sodium hydroxide of approximately 18 to 20% concentration, at elevated temperatures of from 140 to 160° F. and under superatmospheric pressures.

2. A process for the treatment of tar distillates containing tar acids to remove and separate the latter, which comprises subjecting the distillate to the action of an aqueous solution of an alkali comprising sodium hydroxide of approximately 18 to 20% concentration, at an elevated temperature of from 140 to 160° F.

3. A process for the treatment of coal tar containing tar acids to remove the latter, which comprises subjecting the said coal tar to the action of an aqueous solution of sodium hydroxide of approximately 18 to 20% concentration at an elevated temperature of from 140 to 160° F.

4. A process for the treatment of tar fractions containing tar acids to remove the said tar acids and prevent emulsification thereof, which comprises subjecting the said tar fractions to the action of an aqueous solution of sodium hydroxide of approximately 18 to 20% concentration at elevated temperatures of from 140 to 160° F. and under superatmospheric pressures.

5. A process for the treatment of tar fractions containing tar acids to remove and separate the latter, which comprises subjecting the said tar fractions to the action of an aqueous solution of sodium hydroxide of approximately 18 to 20% concentration at an elevated temperature of from 140 to 160° F.

6. A process for the treatment of tars containing tar acids to remove and separate the latter which comprises subjecting said tars at temperatures within the range of 140 to 160° F. to the action of an aqueous solution of caustic soda of approximately 18 to 20% concentration.

7. A process for separating tar acids from tar fractions containing the same which comprises treating the tar fractions at from 140 to 160° F. with aqueous caustic soda of approximately 25° Baumé strength.

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