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UPGRADING COAL TAR

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The present invention relates to the upgrading of coal tar and coal tar pitch.

Coal tar and coal tar pitch contain as their least fusible or volatile component a small percentage, e.g., 2–13%, of quinoline insolubles. This material is the insoluble residue left after extraction of the coal tar or pitch with a large excess of quinoline at 80° C. and comprises primarily coal dust, coke breeze, inorganic matter, graphitic carbon, amorphous carbon and organic high polymers and macromolecules. It is of little if any value in the coking process for the preparation of electrodes and is undesirable in other uses of pitch such as in protective coatings.

Coal tar and coal tar pitch contain a larger percentage, usually 5–22% of a material insoluble in a large excess of benzene at 80° C. The benzene insolubles are generally about double the quinoline insolubles and include in addition to the quinoline insolubles certain organic polymers and macromolecules which on the average have a lower molecular weight than the organic polymers and macromolecules present in the quinoline insolubles.

The part of the benzene insolubles that is soluble in quinoline is of value in most of the uses of pitch and is of great value in one of the principal uses of pitch, namely in making electrodes for the aluminum and other electrochemical industries. It adds to the coking value of the pitch and produces more and stronger coke therefrom.

It is an object of the present invention to prepare a coal tar or coal tar pitch free from quinoline insolubles but containing the remaining components of the benzene insolubles.

Another object is to provide an improved procedure for separating the benzene insoluble fraction of coal tar pitch which is soluble in quinoline from the quinoline insoluble fraction of the pitch.

A further object is to provide an improved procedure for preparing a coal tar pitch residue which is of improved value in the manufacture of high purity graphite, electrodes and activated carbon.

Still further objects and the entire scope of applicability of the present invention will become apparent from the detailed description given hereinafter; it should be understood, however, that the detailed description and specific examples, while indicating preferred embodiments of the invention, are given by way of illustration only, since various changes and modifications within the spirit and scope of the invention will become apparent to those skilled in the art from this detailed description.

It has now been found that these objects can be obtained if coal tar or coal tar pitch is treated with benzene at superatmospheric pressure. For best results the treatment is carried out at a temperature of 120 to 200° C. and at a pressure of 35 to 300 p.s.i.g.

When benzene is mixed with coal tar the benzene dissolves most of the components of the coal tar. Thus, immediately miscible with the benzene are the liquid hydrocarbons such as toluene, the xylenes, styrene, indene, naphthalene and methyl naphthalenes. Also miscible are the tar bases present such as pyridine, quinoline, isoquinoline and their homologues. As a residue which does not dissolve there remains the benzene insolubles. However, by the use of superatmospheric pressure, as set

forth above, the benzene coal tar mixture becomes an even better solvent and dissolves the organic macromolecules and polymeric organic compounds present in the benzene insolubles and even dissolves some of the quinoline insolubles, leaving behind as a residue only such materials as coke breeze, graphitic and amorphous carbon and inorganics.

After separation of this residue the benzene is removed from the extract and the benzene free extract is further distilled to leave behind a pitch residue which is unusually valuable both for high purity graphite, for electrode manufacture and for the preparation of activated carbon because it is completely ash free and also is free of the quinoline insoluble carbon.

In place of pure benzene there can be employed solvents such as light oil which contains a major amount of benzene. Light oil is defined in Von Lauer Patent 2,346,-524 on page 1, column 2, lines 3–18.

Coal tar as is well understood in the art contains coal tar pitch admixed with lower boiling materials and when reference is made in the instant specification and claims to coal tar it is generic to the use of coal tar pitch unless a contrary meaning is clearly indicated.

Unless otherwise stated all parts and percentages are by weight.

While the specific example is directed to a batch procedure the present invention can be carried out in continuous fashion. Thus benzene or light oil can be introduced with coal tar into a centrifugal pump and the mixture forced under turbulent flow through a pipe in a heating zone to a pressure filter. The filter can be stationary and operated on a cycle comprising solution of the coal tar, solvent wash and cleanout, or it can be a continuous rotary filter providing for wash and discharge of solids.

Example 1

1000 parts by weight of coal tar having a benzene insolubles content of 19% and a quinoline insolubles content of 12% were placed in an autoclave with 1200 parts by weight of benzene. The water present in the coal tar (3%) was removed by azeotropic distillation, returning the benzene. After removal of all of the water, the mixture was heated to 158° C. at 80 p.s.i.g. Then the mixture was filtered through a pressure filter and the residue washed with benzene until clean. (The wash liquor was saved for direct use on a second batch of coal tar without intermediate purification.)

The benzene in the extract was removed by fractional distillation at atmospheric pressure, making a cut at 90° C., which is slightly above the boiling point of benzene. The fractional distillation procedure was continued up to a temperature of 350° C. at 10 mm. The residue from the fractional distillation was the mixture of organic high polymers and macromolecules free from ash and quinoline in soluble carbon and amounted to 17% of the original coal tar.

In place of fractionating the purified coal tar by distillation, it can be fractionated by further selective extraction by solvents such as hexane, for example.

Example 2

Example 1 was repeated but the mixture was only heated to 120° C. The pressure rose to about 35 p.s.i.g. The liquid was filtered under pressure and the residue on the filter washed with hot benzene. 61 parts of "quinoline insoluble" material were thus removed by filtration. The remaining liquid was stripped free of benzene to yield a high quality tar suitable for producing ash free pitch coke of particular value for high quality graphite manufacture.

Example 3

The process of Example 1 was repeated utilizing 1000

parts of coal tar and 900 parts of benzene. The mixture was heated to 200° C. and the pressure was approximately 300 p.s.i.g. There was obtained 13 parts of a high quality tar.

We claim:

1. A process for removal of the inorganic components, and quinoline insoluble carbon from coal tar comprising extracting the coal tar with benzene at a temperature between 120 and 200° C. and superatmospheric pressure of 35 to 300 p.s.i.g.

2. A process of fractionating coal tar comprising heating the coal tar with benzene at a temperature of 120 to 200° C. and at superatmospheric pressure of 35 to 300 p.s.i.g., separating the soluble fraction from the insoluble fraction and separating from the soluble fraction that portion which is insoluble in benzene at atmospheric pressure.

3. A process comprising mixing benzene with coal tar, removing the water from the coal tar by azeotropic distillation, heating the mixture to 158° C. at 80 p.s.i.g. and separating the insolubles from the mixture.

4. A process according to claim 3 wherein after separating the insolubles, the mixture is further fractionated by distillation and the undistillable residue is recovered.

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