

UNITED STATES PATENT OFFICE

2,093,450

METHOD OF PRODUCING BODIES OF BITUMINOUS OR TARRY MATERIAL

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No Drawing. Application September 29, 1934,
Serial No. 746,147. In Germany October 30,
1933

16 Claims. (Cl. 196—74)

The invention relates to a method of producing bodies of bitumen and tar, in particular for roads and underground construction. It is known to reduce the surface tension with respect to stone of tar- and bitumen-bodies which are used in the construction of roads and as insulating materials for waterproofing underground and foundation structures, by additions of various kinds, for example by distillation products from fossil products, which contain to a considerable extent bases with a strong nitrogen content.

In the use of such nitrogen-containing substances, acting as covering media use is always made of bodies of a different nature. As the result of systematic research it has been ascertained that not only the covering power but also the binding power of bituminous masses of every kind can be increased without the addition of foreign substances. It is immaterial whether the tar- and bitumen-mass is used in the form of an emulsion or diluted with solvents.

The increase in the covering- and binding-power is obtained by treating the materials to be improved in their technical properties at comparatively not too high temperatures with air or oxygen and at the same time adding a small amount of sulphuric acid of .2% to 3.0% by weight, which has a concentration of 66° Baumé. With this treatment high molecular carboxylic acids, hydroxycarboxylic acids and also sulphur-containing acids are preferably formed. In addition to these by internal ring formation and by polymerization lactones are also formed. The resulting compounds may have a considerably greater molecule than the substances which normally form asphalt- and pitch-masses. Of these substances in particular carboxylic acids, hydroxycarboxylic acids and sulphur-containing acids increase the covering-power of the bituminous products in considerable quantities and form in part true salts with the stone constituents, for example the carbonates and oxides. In addition to such surface actions the intensive binding properties of the lactone, polylactone and the high molecular hydrocarbons produced by continued conversion become particularly effective.

The simultaneous reaction of sulphuric acid and air on petroleum asphalts has already been proposed, but here considerably higher temperatures were used, to produce products of particularly high melting point. Due to the greatly increased temperature complete decomposition took place of the added sulphuric acid—used in larger quantities—and separation of the carboxyl group from the carboxylic acids and hydroxycarboxylic

acids occurred, while in accordance with the invention the additive compounds from constituents of the starting materials and of the 66° Baumé sulphuric acid and the organic acids and oxy-acids formed for the most part remain and ensure that the road binding media produced coat the stone used in the road construction well and thereby firmly bind it, particularly in the presence of the high molecular compounds formed by internal ring formation.

As another advantage it is to be considered that it is possible to employ an asphalt or tar product, which at first is in a freely flowing condition and then forms without hardening too quickly in the air, a very reliable, thin and uniform coating on the stone material, which coating is subjected to a gradual chemical change. The products may accordingly serve both for the surface treatment of the road as also—and this is technically particularly important—for coating road materials, such as broken stones, chips or the like, which are stored and laid on the road at a suitable time.

A further advantage of the process consists in this that with the use of a relatively very liquid bitumen oxidized by the blowing process one is independent of the weather. Even damp stone material is particularly satisfactorily coated, since then the formation of salts is somewhat accelerated, the oxidized masses producing an extraordinary and unforeseen penetrative effect. The effect can be increased if mixtures of tars and asphalts are used, and it is immaterial what kinds of tars are used to provide the mixtures before or during the oxidation process. The method of producing is also applicable to shale and brown-coal-tar. With coal tar it is desirable that it should be as free as possible from naphthalene, since this has often proved unsatisfactory on the road and for foundation work. With material containing paraffin, for example tars from brown coal, the oxidation should be carried out for a longer period and the acid addition somewhat increased but generally even in this case 3% is sufficient.

In the further course of this work the quite surprising result has been found that a particularly advantageous effect is obtained if, in lieu of starting with a mixture of the residues obtained by separate distillation, the raw tars are distilled mixed together or with petroleum and the residues produced subjected to the described treatment. Preferably said petroleum have a high sulphur content.

Quite extraordinary and unforeseen advantages have thereby been obtained. In the first place

the distillation process need not be carried so far as would be necessary to obtain from coal tar alone a soft pitch or from petroleum alone an even lower melting asphalt. Consequently much higher outputs are obtained, since a much greater quantity of the starting material can be used for the desired purpose. This important fact very probably depends on this that particularly the unsaturated compounds of the starting materials of different nature react in the simultaneous distillation with one another and by addition, ring formation, condensation and/or polymerization produce increases in size of molecules and in viscosity, which can only be attained with the separate tars or petroleum after much longer distillation.

The residues of the simultaneous distillation especially possess a greater covering- and binding-action than apparently similar mixtures of "residues" obtained by separate distillations of tars and petroleum. In particular it has been found that the mixed residues produced in accordance with the invention on treatment with air or oxygen and the simultaneous action of sulphuric acid and other oxidizing and/or polymerizing substances produce much more quickly and at much lower temperature the desired end products, the covering- and binding-power of which is greater than with correspondingly treated mixtures of residues from separate distillations.

In all cases further improvements in the production and for different purposes may be obtained by a small addition of fatty oils, resin soaps, peroxides, or materials, which are used as drying agents in the paint industry and have an equivalent effect thereto.

All products produced in accordance with the described process can also be used either in the form of aqueous emulsions or diluted with solvents.

In explanation of the operation of the process the following examples will suffice:

Example 1

	Kilograms
Petroleum asphalt.....	700
Shale tar.....	200
Coal tar-soft pitch.....	100

or only one of these substances are heated to 248° F. with stirring with dry steam. When the temperature is reached, the steam is gradually replaced by air. Steam is added only in such quantity as is necessary to prevent excessive increase in temperature and minimize danger of fire. The issuing vapors are returned or condensed in suitable coolers and obtained as distillate. At about 266° F. 2-10 kg. sulphuric acid having a concentration of 66° Baumé are added slowly. Part of the acid escapes after reduction as sulphurous acid. When the product is brought to the desired physical condition by oxidation and polymerization and internal ring formation, the air is shut off and the substance itself, or in the form of an emulsion or diluted with solvents, used as road-construction or insulating- and packing-material.

Example 2

	Kilograms
Coal tar-soft pitch (prepared tar).....	700
Petroleum asphalt.....	200
Brown-coal-tar pitch.....	100

are mixed with 1-10 kg. linseed oil and 0.5 kg. manganese borate or 0.2 kg. cobalt oxide or litharge, the last mentioned addition being suitably ground previously with fatty oil. The mixture is, as in the first example, heated to 248° F.

and blown with air under addition of about the same quantity of 66° Baumé sulphuric acid and small quantities of persulphate, perborate, peracids, and the like, until a sufficient degree of oxidation and polymerization is attained.

The duration of the blowing operation can obviously be varied within wide limits, according to the type of starting material and according to the desired consistency of the end product. As a guide it may be mentioned that in accordance with the invention there is obtained after a few hours, for example eight hours, and at temperatures slightly over 266° F. a material of consistency which can be obtained with the usual blowing methods only after 24 hours and at much higher temperatures, without possessing the described valuable covering- and binding-properties. The times are only approximate and not given as limits.

Example 3

A mixture of equal parts of raw coal tar and shale tar after removal of the naphthalene fraction provided a suitable starting material which after 5-6 hours blowing at still lower temperatures and with smaller additions of sulphuric acid or similar acting substances than in the first two examples produced a product of like consistency, but with more valuable physical properties and with higher covering- and binding-power.

Example 4

With mixtures of brown coal tar and petroleum the appropriate starting material is obtained by removal of the solar oil or paraffin oil fraction. In the further treatment of this residue the same advantages were obtained as regards time, temperature and properties as with Example 3.

I claim:

1. A process for obtaining a bituminous product comprising the steps of admixing an amount of .2% to 3.0% by weight of sulphuric acid to a tarry material, and blowing said mixture of a tarry material and sulphuric acid with a free oxygen containing gas at a temperature of about 266° F., said sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

2. A process for obtaining a bituminous product comprising the steps of admixing an amount of .2% to 3.0% by weight of sulphuric acid and oxidizing agents to a tarry material, and blowing the mass thus obtained with a free oxygen containing gas at a temperature of about 266° F., said sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

3. A process for obtaining an emulsifiable bituminous product comprising the steps of admixing an amount of .2% to 3.0% by weight of sulphuric acid to a mixture of different bituminous materials, and blowing the mass of different bituminous materials and sulphuric acid thus obtained with a free oxygen containing gas at a temperature of about 266° F., said sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

4. A process for obtaining a bituminous product comprising the steps of admixing an amount of .2% to 3.0% by weight of sulphuric acid and a small amount of an oxidizing agent to a mixture of different bituminous materials, and blowing the mass thus formed with a free oxygen containing gas at a temperature of about 266° F., said sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

5. A process for obtaining a bituminous prod-

uct comprising the steps of admixing an amount of .2% to 3.0% by weight of sulphuric acid and a small amount of peroxides to a mixture of different bituminous materials, and blowing the mass thus obtained with a free oxygen containing gas at a temperature of about 266° F., said sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

6. A process for obtaining a bituminous product comprising the steps of admixing an amount of .2% to 3.0% by weight of sulphuric acid and a small amount of peracids to a mixture of different bituminous materials, and blowing the mass thus obtained with a free oxygen containing gas at a temperature of about 266° F., said sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

7. A process for obtaining a bituminous product comprising the steps of admixing an amount of .2% to 3.0% by weight of sulphuric acid and a small amount of a salt of a peracid to a mixture of different bituminous materials, and blowing the mass thus obtained with a free oxygen containing gas at a temperature of about 266° F., said sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

8. A process for obtaining a bituminous product comprising the steps of admixing an amount of .2% to 3.0% by weight of sulphuric acid and a small amount of a drying agent to a mixture of different bituminous materials, and blowing the mass thus obtained with a free oxygen containing gas at a temperature of about 266° F., said sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

9. A process for obtaining a bituminous product comprising the steps of admixing an amount of .2% to 3.0% by weight of sulphuric acid to a mixture of different tars, and blowing the mass thus formed with a free oxygen containing gas at a temperature of about 266° F., said sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

10. A process for obtaining a bituminous product comprising the steps of admixing an amount of .2% to 3.0% by weight of sulphuric acid to a mixture of different tars and petroleum, and blowing the mass thus obtained with a free oxygen containing gas at a temperature of about 266° F.,

said sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

11. A process for obtaining a bituminous product comprising the steps of simultaneously distilling a mixture of different tars, admixing an amount of .2% to 3.0% by weight of sulphuric acid to the residues obtained by said distillation, and blowing said mixture of said residues and sulphuric acid with a free oxygen containing gas at a temperature of about 266° F., said sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

12. A process for obtaining a bituminous product comprising the steps of simultaneously distilling a mixture of at least one tar and petroleum, admixing an amount of .2% to 3.0% by weight of sulphuric acid to the residues obtained by said distillation, and blowing said mixture of said residues and sulphuric acid with a free oxygen containing gas at a temperature of about 266° F., said sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

13. A process according to claim 12 in which said petroleum is a petroleum having a high sulphur content.

14. A bituminous product obtained by blowing with a free oxygen containing gas at a temperature of about 266° F. a mixture containing a bituminous material and an amount of .2% to 3.0% by weight of sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

15. An emulsifiable bituminous product obtained by blowing with a free oxygen containing gas at a temperature of about 266° F. a mixture containing a mixture of different bituminous materials and an amount of .2% to 3.0% by weight of sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

16. A bituminous product obtained by blowing with a free oxygen containing gas at a temperature of about 266° F. a mixture containing a mixture of different bituminous materials a small amount of an oxidizing agent and an amount of .2% to 3.0% by weight of sulphuric acid having a strength sufficient to form sulpho acids during the reaction.

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