Patented July 28, 1942 2,291,325

UNITED STATES PATENT OFFICE

2,291,325

METHOD OF INDUCING SOLVENCY AND TOXICITY

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No Drawing. Application July 13, 1940, Serial No. 345,441

4 Claims. (Cl. 196—53)

The present invention relates to the joint production of solvents and wood preserving oils.

More specifically the present invention relates to the joint production of solvents and wood preservatives from high boiling fractions of tars of aromatic content; such high boiling fractions include topped tars, residual tars, high boiling fractions, residues, polymerized fractions, and pitches of various nature. The tars from which said high boiling fractions are recovered are tars of aromatic content and include as an example, tars derived from coal, wood, and petroleum. Such tars include the well known "aromatic tars" of petroleum origin.

An object of the present invention is a joint production of solvents and wood preserving oils of lowered boiling range from high boiling tar fractions of higher boiling range, said conversion being characterized by controlling process variable to the end that said solvents have an aromaticity in the order of 50% or more, and the wood preservatives have a toxicity in excess of their parent material.

As is well known in the art, to produce a solvent having an aromatic content of 50% or more from aromatic tars it is necessary to so coordinate process variables that the aromatic nuclei of the starting material is protected against excessive rupture and subsequent degradation. It is equally well understood that in the provision of a wood preservative from a given starting material wherein said preservative is more toxic than the parent material, it is necessary also to protect the aromatic nuclei of the starting material against excessive rupture and subsequent degradation.

By the process of the present invention, the starting materials are subjected to the action of hydrogen, while contacting a catalyst adapted to influence the decomposition of oxygenated organic compounds contained in said starting material, whereby to enhance the induction of solvent fractions and toxicity.

The starting materials of the present process are substances of a questionable value and it has long been a desideratum in the art to convert said starting materials to products of greater value.

The process of the present invention is characterized by subjecting the starting materials to the action of hydrogen with time, temperature and pressure so controlled that no substantial deposition of carbonaceous increment is induced.

By the phrase "no substantial deposition of carbonaceous increment" is meant that deposition of high carbon material including coke that results from too high temperature, and does not include carbon deposition inherent to the process.

The following examples will serve to illustrate modes of practicing the present invention.

Example 1.—A coal tar residuum characterized by oxygen content and boiling substantially 15% at 355° C. and having a specific gravity in the order of 1.20 was charged to high pressure autoclave and subjected to the action of hydrogen, while contacting a catalyst of molybdenum sulfide and a small amount of iodoform, at a temperature of 385° C. and a pressure of 350 atmospheres for a period of one and one-half hours. The beneficiated material was fractionally distilled to an upper limit of 355° C. to provide the material boiling below 200° C. as a distillate having in the order of 50% aromaticity or more and the portion boiling between 200–355° C. as a wood preserving oil more toxic than its parent material. By controlling the reaction as named no substantial percentage of carbonaceous deposition was noted; and as regards the wood preserving fraction, toxicity was also induced as a result of the joint decomposition of oxygenated compounds as influenced by the presence of said halogen-derived catalyst, and by hydrogenation. The presence of said halogen-derived catalyst influenced the elimination of oxygen and/or hydrogenation of carbon to oxygen bonds, thereby assisting in induction of solvents of substantial aromaticity and wood preserving oils of enhanced toxic value.

Thus, the present invention will be seen to provide a process for the conversion of high boiling tar fractions having little or no solvency, and little or no toxicity, to solvents and wood preservatives; said conversion being effected by the controlled action of hydrogen whereby to produce solvents of good aromaticity and wood preserving oils of enhanced toxic value the while precluding substantial carbon deposition; said controlled hydrogenation being further characterized by being effected in the presence of a catalyst adapted to influence the decomposition of oxygenated compounds contained in starting material.

There is no inflexible point of fractionation between the solvents and the wood preserving oils of the present invention, in as much as solvents currently on the market have varied end points, and wood preserving oils of commercial acceptance have varied initial boiling points.

In the fractionation of the beneficiated material, the end point of the solvent substantially

corresponds to the initial boiling point of the wood preserving oil of induced toxic characteristics.

As illustrating this, the tubular data shown below are the end points of commercially accepted solvents, and the initial boiling points of commercially accepted wood preserving oils.

Commercial solvent:	End point, °C.
Benzol	120
Toluol	150
High flash naphtha	200
Heavy naphthas	Above 200

Commercial wood preservative:	I. B. P., °C.
American Wood Preservers' Association	5% at 210
American Wood Preservers' Association	1% at 210
American Wood Preservers' Association	1½% at 235
Prussian Ry	3% at 150
Neville Shingle Stain Oil	150
Southern Pine Shingle Stain Oil	92

Example 2.—An aromatic tar fraction boiling substantially 3% at 380° C. was passed through a high pressure reaction vessel, while contacting a catalyst consisting of molybdenum sulfide and tin chloride, at a pressure of 325 atmospheres and a temperature of 385° C., while flowing with said tar fraction 20,000 cubic feet hydrogen per barrel feed stock. Time of contact substantially one hour. The beneficiated material was fractionally distilled to an upper limit of 375° C. to recover the portion distilling up to 235° C. as a solvent having an aromaticity in the order of 50% or higher, and the portion distilling between substantially 235° C. and 375° C., as a wood preserving distillate of enhanced toxic value.

Example 3.—A topped tar boiling substantially above 250° C. was passed through a high pressure reaction vessel while contacting catalytic material comprising cobalt sulfide and tin chloride, and while passing therewith eleven thousand cubic feet hydrogen per barrel feed stock. Pressure 400 atmospheres, temperature 405° C., time of contact 1½ hours. The beneficiated material was distilled to an upper limit of 270° C. to provide the distillate as a solvent having an aromaticity near the order of 50% or more, with the residue therefrom serving as a substitute for wood preserving oils of the carbolineum boiling range, the fraction above 270° C. for use as a wood preserver being characterized by a toxicity in excess of a comparable boiling out from its parent material.

All catalysts effective in the presence of hydrogen for aforesaid purposes are usable in conjunction with catalysts of halogen origin. As for instance, chromium, molybdenum, vanadium, uranium, cobalt, copper and their compounds as sulphides or oxides; promoted or not; with or without small amounts of alkali, acid or halid, or derivatives thereof. Small amounts of halogen or halogens, may be used as such, or incorporated with other substance; or effective catalysts deposited on carriers, as for instance gels, earths, carbon, or the like. The catalyst may be in various shapes, as for instance forms, extruded shapes or lengths, pellets, comminuted; or mixed with other materials possessing desired action or not; with or without other material effecting splitting or not; or catalyst with added halogen derivatives.

In the foregoing examples the residues resulting from distillation recovery of a wood preserving oil from the beneficiated tar fractions may be used as an article of commerce or recycled for further treatment in accordance with a process of the present invention.

The starting materials of the present invention are characterized by content of oxygenated compounds; said oxygenated compounds, under the influence of a catalytic material selected from the group consisting of halids, and halogens, and derivatives thereof, and while in the presence of hydrogen, are decomposed whereby to provide enhancement of toxicity induction.

Example 4.—A stripped tar boiling substantially above 240° C. and characterized by content of oxygenated compounds was subjected to the action of hydrogen at 400° C. and a pressure of 350 atmospheres for a period of one and one-half hours. Catalyst, molybdenum sulfide and iodoform. The beneficiated material was fractionally distilled to an upper limit of 360° C. the fraction boiling below 200° C. comprising the solvent of the present invention and having an aromaticity of not substantially less than 50%, with the fraction boiling substantially between 200° C. and 360° C. serving as the wood preservative of the present invention having a toxic value in excess of its parent material. The residue incidental to said distillation may be recycled if desired, or used as an article of commerce of value because of its enhanced properties.

A mention has been made of employing a material selected from the group of halids, halogens, or derivatives thereof, for the purpose of influencing the decomposition of oxygenated compounds contained in the starting material. Said decomposition influencer may be used alone or in the presence of catalyst material adapted to further influence hydrogenation. When using said decomposition influencer alone, said influencer may be added to the starting material in small amounts, as for instance one tenth of one percent or more, as based on the starting material. When said decomposition influencer is used with or incorporated in other catalyst, said influencer may represent as much as 50% of the total catalyst, or more.

When using a starting material of inordinately high carbon content it may be desirable at times to at least partially depolymerize the molecular complexes contained therein by aid of a solvent, and said solvent may be used in small amounts or even in amounts equal to the volume of the starting material, or more.

The preferred temperatures of the process are chosen from the range above 300° C., however, lower temperatures are usable. Pressures of 200 atmospheres and above are preferred, as for instance 300 or 400 atmospheres; however, lower pressure is usable. Using average starting materials, as for instance a soft pitch obtained from coal tar, when employing a temperature of 400° C. and a pressure of 300 atmospheres, in the order of one hour will serve to induce a substantial percentage of the solvents and wood preservatives of the present invention without substantial carbon deposit as aforestated. The time element cannot be stated as an arbitrary figure because of the variation in starting materials and the wide latitude of possible coordinations of pressure and temperature. Thus, the required time is that period necessary to induce the predetermined percentage of said solvent and wood preservative increment when coordinated with

the temperature and pressures chosen to preclude substantial carbon deposit.

By the term beneficiated as used herein and in the appended claims is meant the starting material at least once subjected to the action of hydrogen in accordance with the present process.

Viewed broadly the present invention provides a process for the joint conversion of high boiling tar fractions to solvents having an aromaticity of at least in the order of 50%, and wood preserving oils of induced toxicity, said conversion being characterized by subjecting said starting materials to the action of hydrogen while contacting a catalyst capable of influencing decomposition of oxygenated compounds.

Minor changes may be made without departing from the spirit of the invention. In the claims affixed to this specification no selection of any particular modification of the invention is intended to the exclusion of other modification thereof.

I claim:

1. In the joint production of solvents and wood preservatives of induced solvency and added toxicity the process which comprises: subjecting aromatic tar fractions containing oxygenated compounds and boiling predominantly above 355° C. selected from the group consisting of residues, pitches, residual tars, stripped tars, topped tars and petroleum aromatic tars to the action of hydrogen; precluding substantial rupture and subsequent degradation of aromatic nuclei by coordination of time, temperature and pressure, the while increasing induced solvency and added toxicity by inclusion as catalytic material a sulfide of a metal and iodoform; and dividing the treated material whereby to provide a relatively low boiling solvent having an aromatic content not substantially less than 50%, and a relatively high boiling oil of the wood preserving type boiling above and contiguous to said solvent, said wood preserving type oil having an initial boiling point below 220° C. and being further characterized by no greater percentage of material boiling below 210° C., nor a greater percentage of materials boiling between the initial point and a specified temperature than is permitted by specifications accepted in the trade for a tar-derived wood preservative.

2. The process of claim 1 wherein the oil of wood preserving type is recovered as a distillate.

3. The process of claim 1 wherein the oil of wood preserving type is recovered as a residue.

4. In the joint production of solvents and wood preservatives of induced solvency and added toxicity, the process which comprises: subjecting aromatic tar fractions containing oxygenated compounds and high molecular substances, and boiling predominantly above 355° C. selected from the group consisting of residues, pitches, residual tars, stripped tars, topped tars and petroleum tars to the depolymerizing action of a solvent and thereafter reducing the depolymerized mass with hydrogen; precluding substantial rupture and subsequent degradation of aromatic nuclei by coordination of time, temperature and pressure, the while increasing induced solvency and added toxicity by inclusion as catalytic material a sulfide of a metal and iodoform; and dividing the treated material whereby to provide a relatively low boiling solvent having an aromatic content not substantially less than 50%, and a relatively high boiling oil of the wood preserving type boiling above and contiguous to said solvent, said wood preserving type oil having an initial boiling point below 220° C. and being further characterized by no greater percentage of materials boiling below 210° C., nor a greater percentage of materials boiling between the initial point and a specified temperature than is permitted by specifications accepted in the trade for a tar-derived wood preservative impregnant.

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