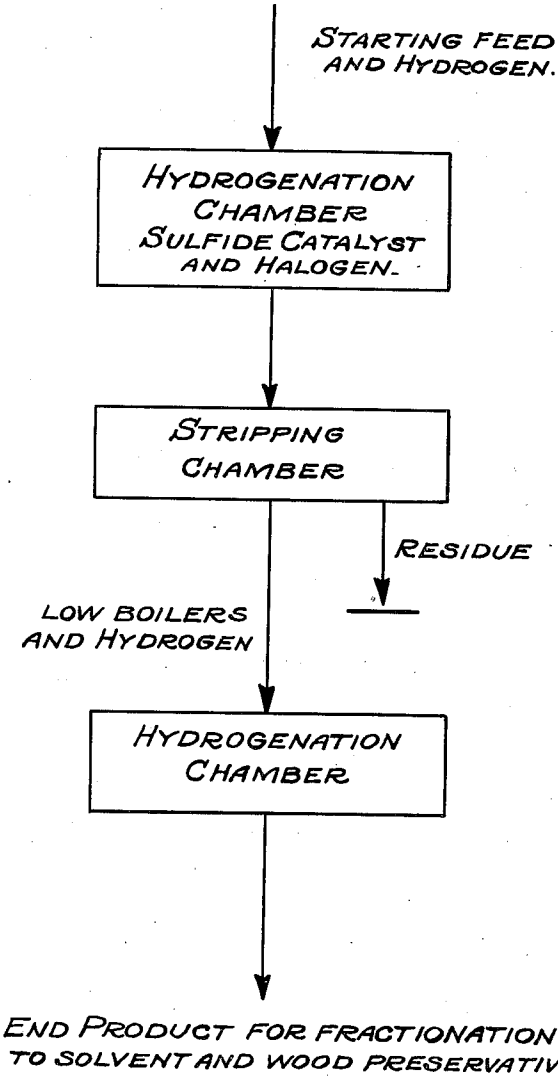
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PROCESS FOR PROVIDING SOLVENCY AND TOXICITY

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PROCESS FOR PROVIDING SOLVENCY AND TOXICITY

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1 Claim. (Cl. 196—53)

The present process relates to the induction of solvency and toxicity.

More specifically the present invention relates to the joint production of solvents and wood preservatives from hydrocarbons.

An object of the present invention is the joint production of solvents and wood preservatives from tars of aromatic content and fractions thereof, said material being characterized by content of oxygenated compounds.

Another object of the present invention is the joint production of solvents and wood preservatives from tars of aromatic content and fractions thereof under conditions that induce no substantial percentage of fractions of low solvency or low toxicity, or, stated in another manner, under conditions so controlled as to induce no substantial percentage of liquid chain structures.

Another object of the present invention is the joint production of solvents of superior solvency and wood preservatives of added toxicity under conditions that induce no substantial percentage of carbonaceous deposition.

Yet another object of the present invention is the enhancing of solvency and toxicity induction by providing means that influence the decomposition of oxygenated compounds contained in the starting material.

Other objects of the present invention will become apparent from the following disclosures.

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

The invention will be understood from the following description of illustrative steps comprising various methods of securing the objects of the invention, when read in connection with the accompanying drawing wherein the figure is a diagrammatic sketch of an apparatus for carrying out a form of the process of the invention and wherein the nature of the step carried out in each chamber and the contents thereof are indicated by legend.

Example 1.—A coal tar creosote, specific gravity 1.08, substantially 2% coke residue and substantially 35% boiling above 355° C. is passed through a high pressure reaction vessel while simultaneously flowing hydrogen therewith, at a temperature of 410° C. and 200 atmospheres pressure. The catalyst is molybdenum sulfide and tin chloride and flow of hydrogen 12,000 cubic feet per barrel creosote and the time of contact 45 minutes. The beneficiated creosote flowing from the reactor is inspected and found to have reduced coke residue, specific gravity, viscosity, and oxygen content. No substantial percentage of liquid chain structures will be induced, nor will there be any appreciable carbon deposit noticeable in the reaction chamber. The beneficiated creosote is distilled to an upper limit of 370° C. and the distillate subjected to the action of hydrogen at 200 atmospheres pressure, flow of 4500 cubic feet per barrel and 450° C., and for such a length of time as to induce solvent properties and toxic increment, said beneficiation being further characterized by a final increment of low boiling fractions in excess of fractional increment in the higher boiling range. The finally beneficiated material is distilled to an upper limit of 200° C. to provide the distillate as a solvent of enhanced solvency and the remainder as a wood preservative of enhanced toxic properties.

By the term "enhanced solvency" or "superior solvency" is meant that the solvent or solvents so described are superior in solvency to solvents currently on the market, also that solvency is induced in the final hydrogen action; by the term "added toxicity" or "enhanced toxicity" is meant that the wood preservative so described has a toxicity in excess of its parent material.

The point of fractionation between the solvent and the wood preservative is not inflexible inasmuch as commercial solvents currently on the market have varied end points and wood preservatives of current usage have varied initial boiling points. Thus, inasmuch as the end point of the solvents of the present invention substantially corresponds to the initial boiling point of the wood preservative, it will immediately be obvious that the point of fractionation is not inflexible, but may be varied at will.

In the tabular data shown below are solvents and wood preservatives of accepted specification, their end point and initial boiling point, respectively:

SOLVENTS

Identification:	End point
Benzol	120° C.
Toluol	150° C.
Hi-flash naphtha	200° C.
Heavy naphtha	Above 200° C.
Plasticizer	160° C. and above

WOOD PRESERVATIVES

Specifications

1. A. W. P. A.
   - *a.* Up to 210° C., not more than 5%
   - *b.* Up to 235° C., not more than 25%
2. A. W. P. A.
   - *a.* Up to 210° C., not more than 1%
   - *b.* Up to 235° C., not more than 10%
   - *c.* Up to 355° C., not less than 65%
3. A. W. P. A.
   - *a.* Up to 235° C., not more than 1½%
   - *b.* Up to 300° C., not more than 16½%
   - *c.* Up to 355° C., not less than 45%
4. A. W. P. A.
   - *a.* Up to 210° C., not more than 8%
   - *b.* Up to 235° C., not more than 35%
5. A. W. P. A.
   - *a.* Up to 210° C., not more than 10%
   - *b.* Up to 235° C., not more than 40%
6. A. W. P. A.
   - *a.* Up to 210° C., not more than 5%
   - *b.* Up to 235° C., not more than 15%
7. Prussian Ry.
   - *a.* Up to 150° C., not more than 3%
   - *b.* Up to 200° C., not more than 10%
   - *c.* Up to 235° C., not more than 25%
8. N. P. V. & L. A. #220
   - *a.* 5% at 162° C.
   - *b.* 97% at 270° C.
9. S. P. S. S. O.
   - *a.* 5% at 137° C.
   - *b.* 95% at 257° C.
10. N. S. S. O.
    - *a.* I. B. P., 150° C.
    - *b.* 5% at 205° C.
    - *c.* 95% at 292° C.
11. Carbolineum, 270° C., I. B. P.

Among other things, the present process is adapted to provide treatment of tars of aromatic content, or fractions thereof, with hydrogen under conditions to decompose at least a portion of oxygenated compounds contained in said tar or fraction thereof, whereby to provide a step in the present process. The final action of hydrogen may be controlled to induce low ends that will substantially correspond to the low ends of any of the solvents noted in the tabular data below:

SOLVENTS

Identification:	Initial point
Benzol	78° C.
Toluol	100° C.
Hi-flash naphtha	150° C.
High boiling crudes	175° C.
Heavy naphtha	150° C.
Plasticizers	160° C. and above

or, solvents of special nature may be produced, as for instance having lower boiling points than above listed.

The foregoing abbrevations are explained as follows: A. W. P. A., American Wood Preservers Association; S. P. S. S. O., Southern Pine Shingle Stain Oil; N. S. S. O., Neville Shingle Stain Oil.

The initial or low boiling point of the beneficiated material is determined by intensity of process controls. Controls of lesser intensity producing higher initial boiling points and controls of greater intensity producing lower boiling points.

Example 2.—A coal tar specific gravity 1.1641 and a coke residue in excess of 5% is passed through a high pressure reaction chamber while simultaneously flowing hydrogen therewith at a temperature of 400° C. and a pressure of 225 atmospheres. The catalyst is cobalt sulfide and iodoform and the flow of hydrogen 12,000 cubic feet per barrel feed stock and time of contact substantially one hour. The beneficiated material is found upon inspection to have lowered coke residue, specific gravity, viscosity and oxygen content. Further inspection will show that no substantial percentage of liquid chain structures have been induced and that the controls of the process have induced no substantial percentage of carbonaceous increment. The beneficiated material is distilled to an upper limit of 330° C. and the distillate subjected to the action of hydrogen while flowing through a reactor at 440° C. and 200 atmospheres pressure. The hydrogen flow is 6,000 cubic feet per barrel feed and the time element is so controlled as to induce solvent and toxic properties; the beneficiated material is further characterized by final increment of low boiling fractions in excess of fractional increment in the higher boiling range. The overall beneficiated material may be used as such or distilled to recover the solvent as a distillate and the remainder thereof as a wood preservative, or, the wood preservative and the solvent both may be recovered as distillates, with a residue therefrom being recycled or serving as an article of commerce, as for instance a binder or plasticizer by virtue of its enhanced value.

The term coal tar in this country is understood to mean tar produced by high temperature carbonization of coal, as for example, high temperature coke oven tar and gas house tar.

Example 3.—A coal tar pitch, specific gravity 1.23 boiling substantially 15% at 355° C. is subjected to the action of hydrogen at 385° C. and 200 atmospheres pressure for a period of one hour. An identical cycle of hydrogen action is repeated on the once hydrogenated pitch; the catalyst in both cycles being tin sulfide and tin chloride. The beneficiated pitch is then distilled to an upper limit of 335° C. and the distillate subjected to the action of hydrogen at a temperature of 465° C. and 200 atmospheres pressure for such a length of time as to induce toxic and solvent properties. The beneficiated material is characterized by a final increment of low boiling fractions in excess of fractional increment in the higher boiling range. The beneficiated material is distilled to an upper limit of 200° C. to recover the distillate as the solvent of the present invention and remainder thereof as a substance having enhanced toxic value which, however, is also usable as a solvent.

The point of stripping the first beneficiated material characterized by reduction of coke residue, specific gravity, viscosity and oxygen content, is dependent upon the end point of the wood preservative desired.

The provision of a catalyst adapted to influence the decomposition of oxygenated compounds contained in the starting material enhances the induction of solvency and toxicity.

The term "pitch" as used herein includes the higher boiling fractions of tars, in other words, tars from which low boiling ends have been stripped, such low boiling ends being suitable per se as creosote, other wood preservative, or solvent. For instance, the final residue resulting from evaporating tar to dryness and then stripping wood preservative from the distillate is a very suitable pitch for use as a starting material of the present process.

An especially attractive mode of practicing the present invention is to provide the cut between the solvent and the wood preservative around 270° C. The wood preservative of the indicated initial boiling point may serve as a substitute for carbolineum wood preserving oils; and the higher boiling portion of the solvent fraction serving as a substitute for certain plasticizing oils.

The residue from any of the distillation steps may be repeatedly recycled for conversion to the products of the present invention. It has been found that the step-wise action of hydrogen has the effect of causing coke residue to substantially disappear.

All catalysts effective in the presence of hydrogen are effective in conjunction with the catalyst which influences decomposition of oxygenated compounds; especially effective are those based on metals of the 6th and 8th periodic groups, as for instance sulfides and/or oxides, separately or in admixture; in any form or shape, as for instance comminuted, pellets, extruded lengths, supported on carriers as for instance on gels or the like. Especially effective are the oxides and/or sulfides of chromium, vanadium, tungsten, cobalt, tin, molybdenum or the like. Other materials effective as splitting agents may be used.

Hydrogen may be supplied as such, or in the form of hydrogen containing gas. Materials capable of supplying or generating hydrogen may be used.

Starting materials are tars of aromatic content or fractions thereof, said tars being derived from coal, wood, petroleum, gas and/or gases, and are characterized by oxygen content. Among such tars are included coke oven tar, gas house tar, a low temperature tar, water gas tar, "synthetic coal tar" of petroleum derivation including gas or gases. Aromatic tars or fractions thereof at least once refined, as for instance by hydrogen, are especially effective starting materials.

Starting materials of the present process also include tars of aromatic content from which low boiling fractions have been removed, as for instance tars from which solvent oils have been removed. Viewed broadly, the starting materials of the present process are tars of aromatic content, fractions of said tar more viscous than the starting material due to removal of low boiling fractions from the starting material, high boiling fractions and pitches.

When using starting materials containing high molecular complexes, said molecular complexes may be depolymerized, or reduced, including in size if desired, until substantially the entirety thereof remaining liquid appears in the solvents and wood preservatives of the present process.

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

It is well known, the action of hydrogen proceeds at lowered pressures, and the present process may be carried on at pressures as low as 50 atmospheres, however, pressures of in the order of 200 atmospheres and thereabove, are preferred. The action of hydrogen in accordance with the present process proceeds at lowered temperatures, however, temperatures of in excess of 300° C. are preferred, the upper limit thereof being defined by that temperature which causes inordinate deposition of carbonaceous increment, as for instance coking.

The time element, because of the possible varied characteristics of the starting or intermediate starting material, cannot be stated arbitrarily; in the first cycle of hydrogen action the time element is that period necessary for reduction of coke residue, specific gravity, viscosity and oxygen content and is generally in the order of one hour. However, using certain feed stocks periods of shorter than one hour have proven effective. Longer periods may be used. In the second cycle of hydrogen action, the period may be as short as one minute, at times more, as for instance several minutes; broadly considered, the time element in the second cycle of hydrogen action is that period necessary for joint induction of solvent and toxic characteristics. Generally speaking, the time element in the second cycle is less than the time element of the first cycle.

The first cycle of hydrogen action, when using certain starting feeds, is characterized by a condensation of the boiling range toward the lower end.

Catalysts adapted to influence the decomposition of oxygenated compounds contained in the starting material are the halogens, halids, and derivatives thereof including substitution and addition products thereof.

Gas flows of in the order of 10,000 to 20,000 cubic feet per barrel feed stock in the first cycle of hydrogen action have proven effective, however, higher or lower flows may be used; in the second cycle of hydrogen action flow of from 6,000–8,000 cubic feet per barrel feed stock has proven satisfactory, however, higher or lower flows may be used.

In the disclosures herein made the removing of low boiling fractions by gas movement or pressure release is considered the equivalent of distillation.

When reference is made to high molecular complexes contained in the starting material, and when the starting material contains low boiling fractions that are not considered high molecular complexes, it is of course obvious that the high molecular complexes contained in the starting material are to a certain extent depolymerized by the solvent present.

A convenient method for testing solvency is by evaluation of the well-known Kauri-butanol number, and toxicity may be evaluated in accordance with "Method of Conducting the Tests," page 2, Technical Bulletin No. 346, March, 1933, U. S. Dept. of Agriculture.

Minor changes may be made without departing from the spirit of the invention.

I claim:

In the joint production of solvents and wood preservatives, the process which comprises: subjecting a mixture of high temperature coal tar fractions to the action of a relatively high flow of hydrogen while contacting a sulfide catalyst and an additional catalyst selected from the group consisting of halogens, halids and derivatives thereof including addition and substitution products thereof; carrying on the process with time, temperature and pressure so controlled as to lower coke residue, specific gravity and viscosity; stripping the beneficiated material at a temperature not substantially in excess of 370° C. and subjecting at least a portion of the stripped low boiling materials in a stream to the action of a relatively low flow of hydrogen not in excess of about 6000 cubic feet per barrel material treated

at a temperature in excess of about 300° C., a pressure in excess of about 50 atmospheres and for such a length of time as to provide fractional increment in the low boiling fractions in excess of fractional increment in the higher boiling range; and dividing the entirety of the treated material into a relatively low boiling solvent and a relatively high boiling oil of the wood preserving type, said oil last named boiling in accordance with specifications accepted in the trade for a tar derived wood preservative.

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