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PROCESS OF MAKING ARTIFICIAL RESIN AND WAX AND THE LIKE FROM COAL

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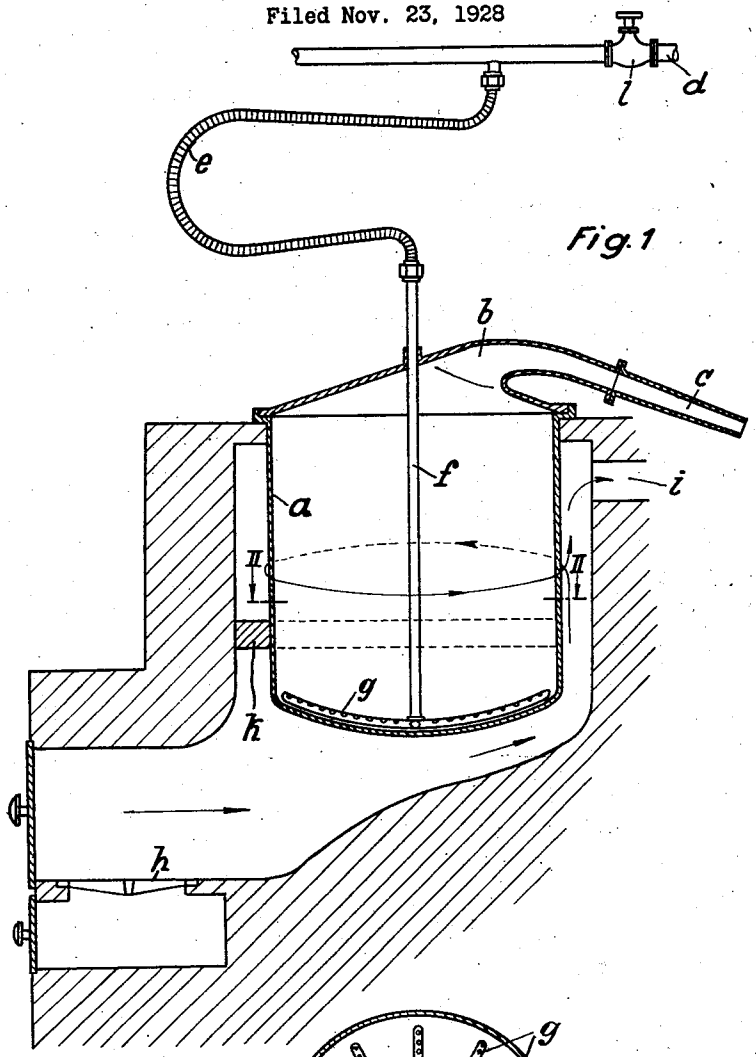

Fig. 1

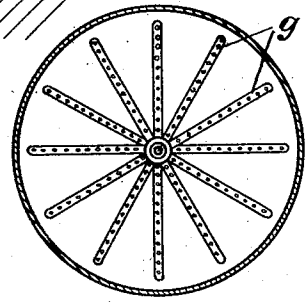

Fig. 2

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PROCESS OF MAKING ARTIFICIAL RESIN AND WAX AND THE LIKE FROM COA

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This invention relates to a process of making artificial resin and wax as well as other hydrocarbons such as crysen, fluoren, pyren, and the like from mineral or brown coal which contains distillable substances such as tar-pitch, by distillation for instance, from molten mineral coal tar-pitch and addition of steam. According to my invention for instance mineral coal tar-pitch, which may either be so-called soft pitch, is molten in a vertical or horizontal retort and thereupon distilled. The same retort may be used for the melting as well as for the subsequent distillation. In order to save fuel the melting process may be carried out in special containers which are heated by the waste heat of the distillation retort. The waste heat more particularly may be used for preliminarily melting the mineral coal tar-pitch. In this case the liquid tar-pitch is caused to flow through a conduit from the containers serving for preliminary heating the tar-pitch into the distillation retort.

In order to increase the yield raw Montan-wax may be added to the molten tar-pitch as well as a catalyst consisting of an intimate mixture of two different inorganic compounds, one being a metallic oxide and the other a halogen salt preferably with the same metallic base as the metallic oxide, so-called contact-substances, which facilitate the re-action. Such mixtures, for instance, may consist of iron-chloride and iron-oxide, aluminium-chloride and aluminium-oxide, calcium-chloride and calcium-oxide, calcium-chloride and aluminium-oxide, sodium-chloride and aluminium-oxide, magnesium-chloride and magnesium-oxide and so forth.

These contact substances during subsequent distillation will bring about an increased production of high-molecular hydrocarbons such as are contained in the completed artificial wax. These additional substances are well distributed in the liquid pitch by stirring.

For the distillation which now follows according to my invention a retort is used which is provided at its bottom with a pipe system for instance of star-shaped conformation, having a great number of small apertures, 350 apertures being for instance used for 1000 kg. of tar-pitch. Through this pipe system steam is introduced into the retort, said steam having approximately a pressure of from 3 to 8 atmospheres. The pipe system is preferably supplied with steam by means of a flexible metal hose and introduced into the liquid pitch.

According to my invention also hydrogen may be introduced into the retort together with the steam in order to produce distillation products of low melting point. In addition to this by employing hydrogen the yield may essentially be increased. The quantity of hydrogen which is introduced together with the steam into the retort may be chosen in dependance from the desired degree of softness of the products to be obtained.

The retort is closed up by a cover which is provided with a pipe-stud serving for connecting said retort to an air cooler in which the distillation products may be condensed.

In order to accelerate the distillation and in order to increase the yield of the products obtained by the process, the distillation may also be carried out in a vacuum. The retort in this case is heated for such a period of time until all volatile constituents of the tar-pitch have been distilled. The yield will vary between 40 and 80% of the original material according to the property of the tar-pitch which is employed as original material.

During the process of distillation at first mostly water steam will be produced during melting of the tar-pitch, said steam originating from the moisture contained therein. Thereupon anthracene oil steam will discharge at a temperature of approximately 240° C. while upon further increase of the temperature to a value above 300 degrees C. there will at first be obtained a yellow wax-like product and thereupon a reddish resin within temperatures of from about 300 to 360 degrees C., these being temperature values as measured at the point where the distillation products discharge from the retort. In the retort itself the temperature will naturally be essentially higher and during the full distillation may reach red incandescence.

During the distillation also ammonia is

produced which may likewise be utilized for practical purpose. The residue in the retort consists of a hard valuable coke which during combustion produces only small quantities of ashes, said coke being, for instance, of value in the electrical industry as well as in connection with processes of melting. The yellow wax and red resin which is gained during distillation may be utilized with advantage in the chemical and related industries. This wax and resin however, may also be molten together with from 3 to 20% refined Montan wax, paraffin or ceresin according to the melting point and the properties desired in the final product, which may be advantageously in the chemical and related industries, especially in the manufacture of varnish, dyes, impregnating materials and the like.

If the distillation products are transformed at definite temperatures, for instance at 150° C., into an alkaline compound, for instance by means of metallic sodium or by means of sodium amide, and thereupon mixed with water free fluorene will be obtained, as a final product.

On the other hand, if the distillation products are treated with carbonic disulphide or another dissolving agent by means of which the insoluble parts may be separated from the soluble parts, the greatest part of the chrysenes may be obtained as a residue of filtration.

In like manner also pyren, fluor-anthen, reten and picen may be gained by the process of distillation.

With the process described in the foregoing large yields may be attained in a commercially successful way. In the following two examples are given for the process according to my invention.

First example (average scale experiment).

26.5 kg. of mineral coal tar pitch are mixed with 300 g. of iron chlorid, 500 g. of iron oxide and 300 g. of Montan wax and treated according to the aforedescribed process. The yield of the process will be 8.4 kg. artificial wax or artificial resin, 2.5 kg. of anthracene oil and 15,5 kg. of coke.

Second example (large scale experiment).

1000 kg. of mineral coal tar pitch are mixed with 5 kg. of iron chloride, 8 kg. of iron oxide and 2 kg. of Montan wax. The yield will be 370 kg. of artificial wax or artificial resin, 80 kg. of heavy anthracene oil and 430 kg. of coke.

In the accompanying drawings I have represented an apparatus for carrying out the process according to my invention. Fig. 1 is a diagrammatic section through a furnace comprising a retort *a* provided with a hood *b*, said hood being connected to a pipe *c* which leads to the condensing apparatus for the vapors produced within said retort *a*. From the steam supply conduit *d* steam is conveyed by way of the flexible metal hose *e* into the retort *a* through the pipe *f* which at its under end is connected to a star-shaped system of pipes *g* as shown in Fig. 2, said pipes *g* being provided with a greater number of apertures as indicated in Fig. 2 of the drawings. The grate of the furnace is indicated at *h*, and the fire gases will take their way as indicated by arrows around the retort *a* to the stack *i* of the furnace, a transverse wall *k* being provided for this purpose around the retort *a*.

The retort *a* is filled with the mineral coal tar, or other original substance hereinabove mentioned, the furnace is heated and steam supplied from the steam conduit *d* with the valve *l* therein to the pipe *f* and the star-shaped pipe system *g*. Steam will thus be passed through the molten mineral coal tar pitch contained within the retort *a*, while the products of distillation will discharge through the pipe *c* and may thereupon be carried into the condensing apparatus.

I claim:

1. In the process of making artificial wax and resin from mineral coal tar pitch, the steps which consist in melting said tar pitch and distilling the same in a retort, passing steam therethrough in the presence of a catalyst composed of a mixture of compounds, one being a metallic oxide and the other a halogen salt preferably with the same metallic base as the oxide, so as to produce in succession a heavy anthracene oil, a yellowish wax and a reddish resin in accordance with the temperature of distillation.

2. A process as specified by claim 1, in which simultaneously with the steam also hydrogen is introduced into the mineral coal tar pitch, the distillation being eventually carried out in a vacuum.

3. A process as specified by claim 1, in which raw Montan wax and contact substances consisting of an intimate mixture of compounds, one being a metallic oxide and the other a halogen salt preferably with the same metallic base as the oxide, are added to the molten mineral coal tar pitch prior to the distillation, and hydrogen simultaneously introduced with the steam, the distillation being eventually carried out in a vacuum.

4. A process as specified by claim 1, in which for the production of a wax-like or resin-like final product the reddish resin as well as the yellow wax produced by the distillation are molten together, refined Montan wax, paraffin or ceresin being eventually added.

In testimony whereof I affix my signature.

EMIL RAHMANN.