

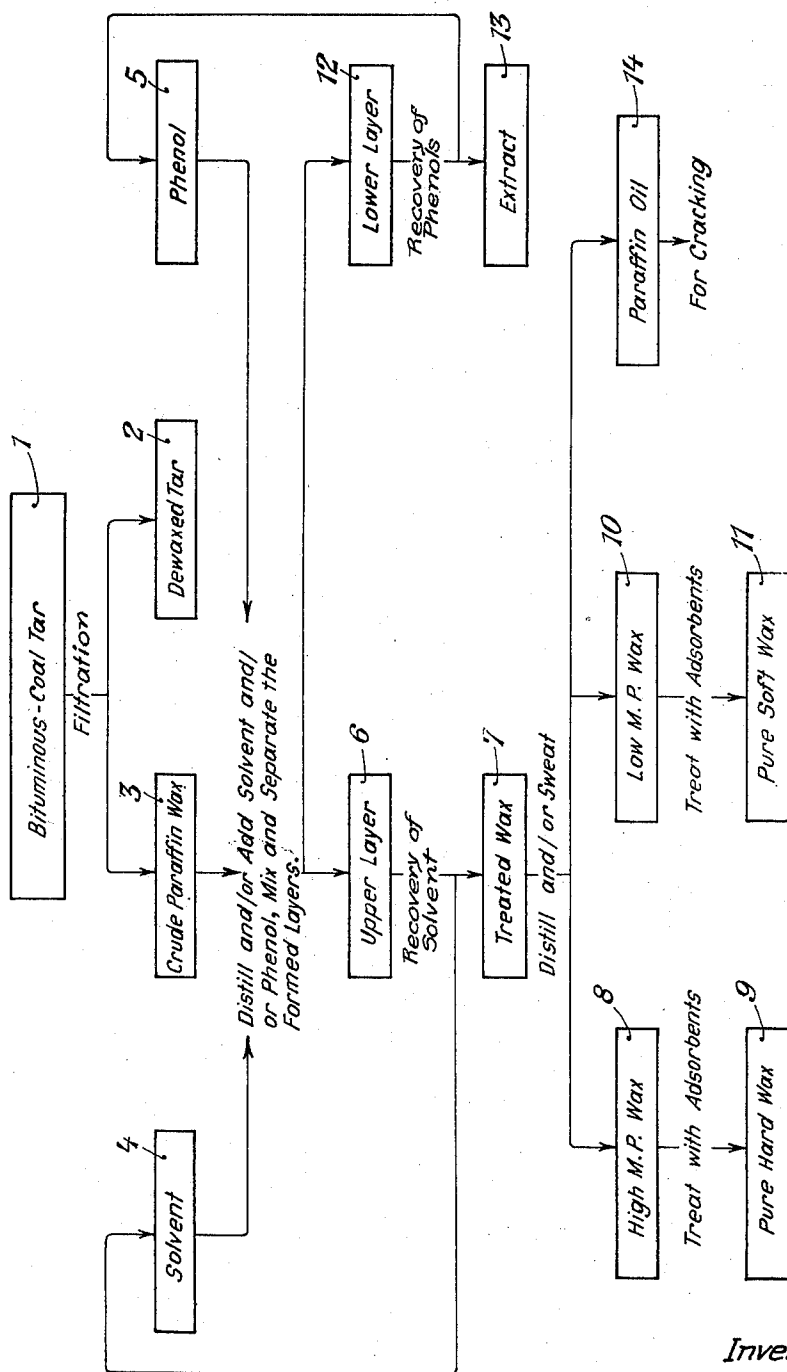
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F. SCHICK

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PROCESS FOR OBTAINING PURE PARAFFIN

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Inventor:

Friedrick Schick

By *H. B. Wilson & Co.*
Attorneys

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PROCESS FOR OBTAINING PURE PARAFFIN

Friedrich Schick, Berlin-Schöneberg, Germany,
assignor to the firm Deutsche Erdöl-Aktiengesellschaft, Berlin-Schöneberg, Germany

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The obtaining of paraffin wax from tars, such as lignite tar, shale tar and peat tar, is effected technically on a large scale, in such a manner that the hotly pressed crude paraffin wax obtained by the filtration of the tar at a temperature of 20° C. (from lignite tar or lignite tar distillate), is freed from creosote by means of soda lye and then subjected to a sweating treatment. The sweated product obtained thereby is treated with sulphuric acid and subsequently with active coal or bleaching earths for the purpose of lightening its colour.

It now has been ascertained that the production of pure paraffin wax can be considerably improved and simplified if the crude paraffin wax is treated in a liquid state with phenols of low boiling point, such as phenol, cresols and xyleneols or mixtures thereof, under certain circumstances. This treatment will cause the phenols of high boiling point which interfere with the sweating process to be dissolved, and simultaneously those impurities of the paraffin wax to be eliminated which in the known process above mentioned require a subsequent refining with sulphuric acid. The necessity of freeing the crude paraffin wax from creosote by treating it with soda lye and refining with sulphuric acid can thus be omitted here. Attention is to be paid in this connection to the fact that the simplification ensured by the new process is connected with the application of a solvent which, just as the paraffin wax itself, is obtained in the working down of lignite tar, whilst for the older processes reagents are required which are to be procured from other and foreign sources only. The refining of the crude paraffin wax by means of phenols suggested by the present process, when compared with the known processes of freeing the crude paraffin wax from creosote, embodies the additional advantage that the sweating treatment for obtaining hard paraffin wax proceeds more smoothly and better and ensures a greater yield.

The new process is carried out in such a manner that the crude paraffin wax while in a thin liquid condition is treated with phenols, an upper layer containing the paraffin wax, and a lower one consisting of the solvent and the dissolved material being formed thereby. The treatment with the phenols is preferably effected in several steps because this will ensure a more thorough separation of the undesirable impurities of the paraffin wax. The phenols may be applied in a concentrated form, or they may also be diluted with water. In some cases it might be advisable to add a solvent for the paraffin wax, such as

benzine, which will facilitate the formation of the layers. The residue which remains undissolved during the extration with phenols is drained off and, after the elimination of the phenols contained therein by distillation or by treatment with steam, is cooled and subjected to a sweating process in a known manner. The product thus obtained after treatment with decolorising agents, for instance with wholly or partly de-ashed semi-coke or low temperature coke is perfectly colourless.

In some cases it will be possible to facilitate the sweating and the brightening of the colour of the crude paraffin wax, if it is distilled under slight cracking conditions before or after its treatment with the phenols. Such a treatment will be particularly advisable, where the tar has been obtained by cautious treatment and the paraffin oil, too, has not been or is carefully distilled.

The run out or discharged oil obtained in connection with the sweating of the raffinate or refined product of the hotly pressed crude paraffin wax may be used as a starting material for thermal cracking processes because it exhibits little tendency to form coke. This proves of special advantage when the cracking is carried through in the presence of contact agents or catalysts.

Example

A paraffin oil obtained by distillation of low temperature tar from Rositz is filtered in a frame filter press at a temperature of about 20° C. The crude paraffin wax or scale obtained thereby is extracted in three steps at a temperature of 60–70° C. with a 90% cresol in quantities of 50% each, referred to the crude paraffin wax. The undissolved material, after treatment or distillation with steam, is substantially free of phenol, and, as a result of the sweating process, yields a sweated product in a quantity of 35.9% with respect to the hotly pressed crude paraffin wax. The paraffin wax obtained after treatment with decolorising semi-coke or low temperature coke has a solidifying point of 52.5° C. and is colourless and odorless.

If the same crude paraffin wax is freed from creosote by means of soda lye in known manner and subjected to sweating, the sweated product thus obtained only amounts to 32.3%, and, in addition to a subsequent refining with bleaching or fuller's earth, treatment with sulphuric acid is necessary.

It has already been suggested to recrystallise or repress paraffin wax in the presence of lignite tar creosotes. This procedure was intended to replace

the sweating process. Furthermore it is known to treat crude paraffin wax with sulphur dioxide at a temperature at which the paraffin is separated as a liquid layer, whereupon the latter is freed from the sulphurous acid by distillation. These processes, however, could not be applied to practical purposes, because the attempts to obtain with their aid a marketable product proved futile.

The above described invention is illustrated in the accompanying diagrammatic flow sheet drawing. Reference is now made to this drawing. The crude paraffin wax 3 which is to be treated may be obtained, for example, from bituminous coal tar 1 by filtration at a suitable temperature, a dewaxed tar 2 being obtained at the same time. From this point, the crude paraffin 3 may be treated in various ways according to the invention, and these are indicated below respectively by the characters (A), (B), (C), and (D). Each of these are described below in the order given. (A) The crude paraffin wax 3 may be treated directly with phenols 5, an upper layer 6 containing the paraffin wax and a lower layer 12, containing phenols and dissolved impurities, being formed thereby. The lower layer 12 is treated to recover the phenols which are again conducted to 5 for re-use, an extract 13 remaining from this recovery. The treated wax 7 obtained from the upper layer 6 after removal of the phenols contained therein, for example by treatment or distillation with steam, is sweated, whereby a high melting wax 8, a low melting wax 10 and paraffin oil 14 are obtained. The latter may be cracked. The waxes 8 and 10 may be treated with decolorizing or bleaching solid adsorbents to form pure hard wax 9 and pure soft wax 11. The phenols may be applied in a concentrated form or they may also be diluted with water. (B) Benzine may be admixed as a solvent 4 to the crude paraffin wax 3 to be treated with the phenols 5, the treatment being the same as described above in (A) except solvent is recovered from the upper layer 6 and returned to 4 for re-use. (C) The crude paraffin wax 3 may be distilled under mild cracking conditions and then treated according to (A) and (B) above. (D) The treated wax 7 obtained according to (A) or (B) above may be subjected to distillation under mild cracking conditions before sweating.

Having now particularly described and ascertained the nature of my said invention, I declare that what I claim is:

1. A process of preparing substantially pure paraffin wax which has a relatively high melting point and which is odorless and colorless, comprising treating crude paraffin wax obtained from bituminous tar with phenols of low boiling point, removing the phenol together with the impurities dissolved therein from the paraffin wax, then sweating the thus treated paraffin wax and finally decolorizing the paraffin wax with an adsorbent carbon.

2. The process as set forth in claim 1 in which the phenol is in an aqueous medium.

3. The process as set forth in claim 1 in which benzine is admixed with the crude paraffin wax.

4. A process of preparing substantially pure paraffin wax from crude wax obtained from bituminous tar of the class consisting of lignite tar, shale tar and peat tar comprising treating the said crude wax with phenols of low boiling point in a series of steps, removing the phenol together with the impurities associated therewith, then sweating the thus treated paraffin wax and finally decolorizing the paraffin wax with an adsorbent.

5. A process of preparing substantially pure paraffin wax from crude wax obtained from bituminous tar which comprises distilling the crude wax under mild cracking conditions, then treating the thus obtained paraffin wax with a phenol of low boiling point, removing the phenol together with impurities associated therewith, then sweating the wax and finally decolorizing the paraffin wax with an adsorbent.

6. The process as set forth in claim 4 in which the partially purified paraffin wax is subjected to distillation under mild cracking conditions after the treatment with the phenol.

7. The process as set forth in claim 5 in which the crude paraffin wax is treated with phenol while the wax is thinly liquid.

8. A process of preparing substantially pure paraffin wax which is odorless and colorless, comprising treating crude paraffin wax obtained from bituminous tar with phenols of low boiling point, removing the phenol together with the impurities dissolved therein from the paraffin wax, then sweating the thus treated paraffin wax and finally decolorizing the paraffin wax with an adsorbent.

FRIEDRICH SCHICK. 50