

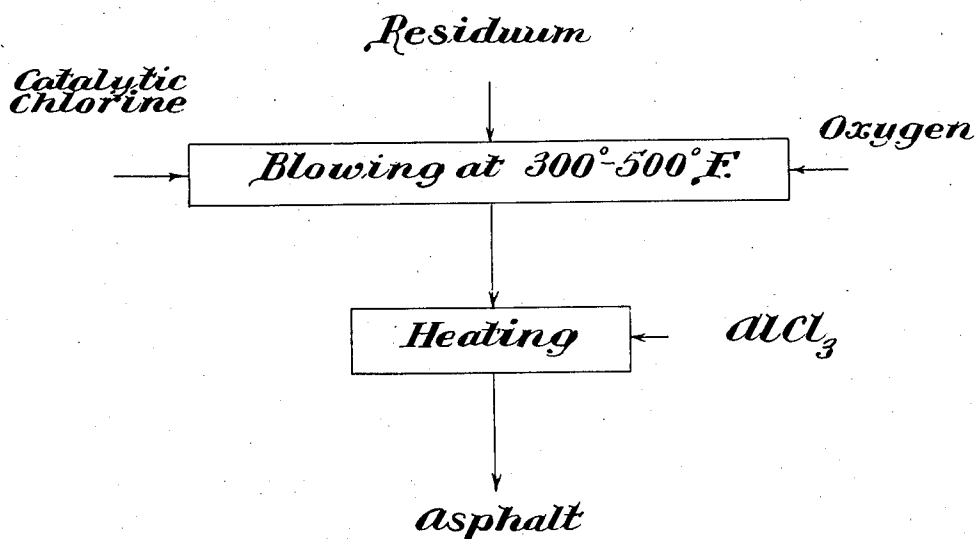
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MANUFACTURE OF IMPROVED ASPHALT

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MANUFACTURE OF IMPROVED ASPHALT

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5 Claims. (Cl. 196—74)

In manufacturing asphalt, it has been the custom to blow the petroleum residuum with air, and maintain the temperature at about 300–500° F. The procedure is very tedious, requiring a long duration of action in order to bring the material to usable characteristics of high melting point and low penetration. Although enormous quantities of oxygen are brought into relation with the hydrocarbon material, there is not a great increase in the oxygen content, and the action is largely a dehydrogenation and conjoining of hydrocarbon molecules. It has not been possible though to attain products with melting point as high as are desirable for a given penetration. In accordance with the present invention however, asphalts of remarkably high melting point for a given penetration may be produced, such products having particular value in usages where exposed to sun and air.

To the accomplishment of the foregoing and related ends, the invention, then, comprises the features hereinafter fully described, and particularly pointed out in the claims, the following description setting forth in detail certain illustrative embodiments of the invention, these being indicative however, of but a few of the various ways in which the principle of the invention may be employed.

The accompanying drawing is a flow sheet illustrating diagrammatically a preferred embodiment of the invention.

In our co-pending application Ser. No. 219,405, filed July 15, 1938, there is set forth the making of asphalt by air-blowing residuum and subsequently treating with a hydro-halogen acid and a halide. In the present process, first the stock to be worked up, a heavy hydrocarbon material containing aliphatic hydrocarbons, for instance a petroleum oil residue, and which we designate residuum, and which with particular advantage may be in some instances a mixture of pipe still bottoms and cylinder oil stock, the latter for example in proportion of 10 to 30 per cent, is heated with oxygen or air, conveniently for instance being blown with air. The temperature of treatment may be 225–450° F., the material being suitably stirred or agitated. And the time of this treatment may be for instance 12 to 24 hours. With the air, or at least during the latter portion of the air-blowing there is carried into the material, most conveniently with the air current, a small amount of chlorine. Having in this manner brought the material up to partial specification, the air-blowing is discontinued, likewise the catalytic chlorine. Then, while maintaining

the temperature elevated, for instance at 300–450° F., a small amount of aluminum chloride, AlCl_3 , is incorporated into the residuum, and is allowed to act for 15 to 60 minutes. The amount of aluminum chloride may be 0.1 to 1.0 per cent, usually 0.25 per cent. In some cases we also add a small amount of a hydro-halogen acid, as hydrogen chloride, (HCl), hydrogen bromide, (HBr), hydrogen fluoride, (HF), hydrogen iodide, (HI), in general the number of mols of hydro-halogen acid not exceeding the number of mols of the halogen catalyst.

Products produced in accordance with the present procedure are asphalts of extraordinarily high melting point with relation to the penetration. These products also have outstanding susceptibility coefficients and have great resistance to oxidation, and are capable of particularly excellent performance in usages such as roofing asphalt, paving filler asphalts, etc., where subjected to particularly drastic exposure to sun and oxidation action. That the action in the present process is not merely the additive effects of oxidation with catalysts and then treatment with aluminum chloride, is shown from comparative tests. Thus, a residuum treated in accordance with customary commercial procedure, by air-blowing to finished specification, shows a melting point of 219°, with penetration 18 at 77° and 31 at 115°. If the same residuum be blown with air and catalytic chlorine, the product comes out with a melting point of 219°, and penetration 13 at 77° and 26 at 115°. If similar residuum be treated with aluminum chloride alone in the small amount contemplated by the present invention, and for comparable time, the product comes out with a melting point of 222°, and penetration 20 at 77° and 37 at 115°. But, if the residuum be air-blown and catalytic chlorine be supplied, and then the air-blowing be discontinued, and 0.25 per cent of aluminum chloride be incorporated in accordance with the present invention, the product comes out for instance with a melting point of 241° and penetration 11 at 77° and 20 at 115°. Melting point relations heretofore unobtainable thus become possible.

This application is a continuation, in part and as to common subject matter, of our application Ser. No. 112,348, filed November 23, 1936.

Other modes of applying the principle of the invention may be employed, change being made as regards the details described, provided the features stated in any of the following claims, or the equivalent of such, be employed.

We therefore particularly point out and distinctly claim as our invention:

1. A process of making asphalt, which comprises air-blowing a residuum and introducing a small amount of chlorine, then discontinuing the air-blowing and introducing into the residuum 0.25 per cent of aluminum chloride and a small amount of a hydro-halogen acid and maintaining the temperature elevated.
- 10 2. A process of making asphalt, which comprises oxidizing a residuum and introducing a small amount of chlorine during at least the latter part of the oxidizing action, then discontinuing the aforesaid and introducing into the
15 residuum a small amount of aluminum chloride and a small amount of a hydro-halogen acid and subjecting the residuum to the action thereof at elevated temperature.
- 20 3. A process of making asphalt, which comprises air-blowing a residuum and introducing a small amount of chlorine, then discontinuing the air-blowing and introducing into the residuum 0.25 per cent of aluminum chloride and maintaining the temperature elevated.
4. A process of making asphalt, which comprises oxidizing a residuum and introducing a small amount of chlorine during at least the latter part of the oxidizing action, then discontinuing the aforesaid and introducing into the residuum a small amount of aluminum chloride and
10 subjecting the residuum to the action thereof at elevated temperature.
5. A process of making asphalt, which comprises air-blowing a residuum and introducing a small amount of chlorine, then discontinuing the
15 air-blowing and introducing into the residuum a small amount of aluminum chloride and maintaining the temperature elevated.

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