Patented Sept. 27, 1927. 1,643,521

UNITED STATES PATENT OFFICE.

CHARLES S. REEVE, OF GRANTWOOD, NEW JERSEY, ASSIGNOR TO THE BARRETT COMPANY, A CORPORATION OF NEW JERSEY.

PLASTIC COMPOSITION.

No Drawing. Application filed January 24, 1922. Serial No. 531,468.

This invention relates to bituminous compositions of various consistencies and rubbery character which may serve a variety of purposes such as paving and roofing compounds, water-proof paints and coatings, rubber compounds, floorings, etc. It deals particularly with a means of producing such compositions from oil shale.

A well known characteristic of tar pitches, such as those obtained as residuals from the fractional distillation of coal tar and water-gas tars, is their relatively high susceptibility to changes in temperature. In other words, a given pitch may vary in consistency from a brittle solid at freezing temperature to a viscous fluid at extremely high summer temperatures. Residual asphalts obtained by the distillation of petroleum also possesses this characteristic to a great extent, but with them, the susceptibility is materally reduced by "blowing" or passing a required amount of air thru them at high temperatures. Such treatment is, however, relatively ineffective on tar pitches on account of the different properties of the hydrocarbons contained in such pitches as compared with those in petroleum residuals. For many purposes, it is desirable to have a bituminous material that is, relatively, only slightly susceptible to temperature changes, in other words, a material that will show a relatively slight change in consistency in passing thru the temperature range to which it will be exposed. A rubbery or elastic character is an additional desideratum which tar pitches do not possess in that a slab of the material when bent or twisted will not of itself straighten out or recover its original form.

I have discovered that compositions of matter possessing these desirable characteristics may be prepared by digesting oil shales with high boiling tar distillates in a manner hereinafter described. These shales consist essentially of a mineral matter, usually argillaceous, and a combination of hydrocarbons, commonly termed Kerogen, which latter upon heating to temperatures which differ somewhat with shales from different localities, is converted into bitumen. If the heating of these shales is carried out in a retort, it is the common practice to carry the temperature beyond the point of formation of bitumen, in which case the bitumen is destructively distilled with the production of shale oil, which may be later distilled and refined for the production of various grades of oils and wax. The purpose of my invention is accomplished by holding the shale at or near the temperature at which the Kerogen is converted to bitumen while being agitated with a high boiling tar distillate.

The following is given as a specific example illustrative of my invention, altho it is understood that I may use a great variety of oils and shales and that I may vary the proportions, time and temperature of treatment to produce the desired product without departing from the spirit of my invention.

I take approximately 1 part of a coal tar distillate, preferably that portion recovered from coal tar after the anthracene fraction has been removed, and 1 part of a Nevada oil shale having approximately 40% volatile matter (American Society for Testing Materials Standard Method D—22—16). The shale is preferably crushed to a size that will pass a 10 mesh sieve altho it is possible to accomplish my purpose with larger size particles. The materials in the above proportions are placed in a metal still or kettle provided with a mechanical stirrer or agitator and a condenser with a receiver for any small amount of distillate that may be evolved, heated to a temperature of approximately 300° C. to 400° C., preferably about 310° C. to 320° C. and maintained at that temperature for 10 to 15 hours while being continually stirred. I have found this optimum temperature to vary with different shales. Some Colorado shales for instance, would require a temperature of 340–350° C. for similar results. At the end of this period, it will be found that the shale has decomposed as evidenced by the fact that the particles have lost their shape that the mineral matter has gone into a state of very fine dispersion with practically no evidence of grit, and that the bitumen formed has entered into combination with the oil. The product may be rendered more homogeneous if desired by passing between rolls.

This product has a very low susceptibility

to temperature changes, resembling partly blown asphalts as shown by the following penetration tests (A. S. T. M. D—5—20).

Penetration (0° C., 200 gms., 1 min.)- 6.5
Penetration (25° C., 100 gms., 5 secs.)- 44.0
Penetration (46.5° C., 50 gms., 5 secs.)- 110.0

While soft and flexible at ordinary temperatures, I have been unable to render it liquid by heating it to a temperature of 500° F. in the regular cube in air test. (Jour. Ind. and Eng. Chem., Oct. 1918.) I know of no other products of either coal tar distillates or oil shales possessing these characteristics.

Another characteristic of compounds prepared by my process is their elasticity or resiliency as compared with coal tar pitches. The former when bent or stretched tend to immediately assume their original shape which is a characteristic entirely absent from the latter.

Compositions of the character described may have a variety of uses. They have the property of strongly adhering to iron and when thinned with solvent may be used as bituminous paints. Their relatively low susceptibility to temperature changes and strong adhesive qualities make these compositions particularly adapted for uses where there would be a tendency for other bituminous materials to flow. The same qualities render the aforesaid compositions particularly applicable as paving joint fillers of the pre-moulded type. They may be renderd fluid by means of a volatile solvent and then used as a saturant for fabrics. In this connection they may prove especially well adapted to fabric belt saturation where pliability at low temperatures and no fluidity at moderately high temperatures is desirable.

In general the products find usefulness in all fiields where bituminous materials are applicable, and the variations of proportions of shale and tar products, the temperature and time of digestion and the final melting point of the product are all within the skill of the chemist familiar with bituminous materials, depending on the purpose for which the final product is to be used.

I claim:

1. A new and useful composition of matter comprising coal tar oil and substantially all of the decomposition products of an oil shale that has been decomposed while in intimate contact with said oil.

2. A new and useful composition of matter comprising coal tar oil having a boiling point above the boiling point of anthracene and substantially all of the decomposition products of an oil shale that has been decomposed while in intimate contact with said oil.

3. A new and useful composition of matter comprising coal tar oil and substantially all of the decomposition products of an equal part of oil shale that has been decomposed while in intimate contact with said oil and in which the mineral matter of the oil shale is finely divided.

4. The process of manufacture of a product containing bituminous matter, which comprises heating oil shale in the presence of coal tar oil at a temperature at which the kerogen is decomposed without permitting substantial distillation of the mixture.

5. The process of manufacture of a product containing bituminous matter, which comprises heating oil shale in the presence of coal tar oil at a temperature of approximately 310° to 320° C. for about 10 to 15 hours.

6. The process of manufacture of a product containing bituminous matter, which comprises heating oil shale in the presence of an equal part of coal tar oil at a temperature of about 315° C. for about 10 to 15 hours.

7. The process of manufacture of a product containing bituminous matter, which comprises heating oil shale in the presence of coal tar oil at a temperature at which the kerogen of the oil shale is decomposed without permitting substantial distillation of the mixture, said coal tar oil having a boiling point above the boiling point of anthracene.

8. The production of a bituminous composition, which comprises digesting oil shale with coal tar oil at such a temperature that decomposition of said shale and the combination of its decomposition products with said oil occur simultaneously without permitting substantial distillation of the mixture.

In testimony whereof I affix my signature.

CHARLES S. REEVE.