Patented Dec. 15, 1942 2,304,773

UNITED STATES PATENT OFFICE

2,304,773

BITUMINOUS PRODUCT AND METHOD OF MAKING

Benjamin Albert Anderton, Cliffside Park, N. J., assignor, by mesne assignments, to Allied Chemical & Dye Corporation, a corporation of New York

No Drawing. Application July 16, 1938, Serial No. 219,645

6 Claims. (Cl. 106—278)

This invention relates to tar products of a novel type adapted for use as roofing pitches upon steep roofs.

Coal-tar pitch has outstanding waterproofing qualities, is immune to climatic variations, is self-healing and is technically "toxic" to fermentation or decay.

The use of coal-tar pitch, however, has been limited to relatively flat decks, i. e. dead level to inclines of less than two or three inches to the foot. This was due to the relative susceptibility of coal-tar pitches to temperature change. Thus, for steep work, and where a form of built-up roofing was desired, it has been expedient to resort to the use of so-called high melting point asphalts, or "bitumens" containing inert fillers.

Originally, built-up roofs were designed for relatively flat decks. Later, as structural trends and necessity gave impetus to industrial steep-roofed construction, the economy of built-up roofs suggested their use in this field.

In the early days saturated burlap or felts were fabricated into a membrane and were given a finishing coating of tar or tar paint sprinkled with sand for use on steep roofs. Then came the pitch and tar job mixtures, to which, later, were added inert fillers, lime, etc. These mixtures might be termed experiments which, with the advent of cracking and blowing processes in the petroleum industry, gave way to petroleum residual asphalts. These asphalts were plentiful and relatively inexpensive, and because of their relative non-susceptibility to slide, required no job mixing or filler. Frequently, however, such asphalts are not uniform, and because of their relatively high softening point range are difficult to heat, are relatively highly viscous when molten and consequently hard to spread. Thus, premium asphalts were developed to inherently lower melting points or "cut back" with oils to facilitate heating and spreading; inert fillers were commonly added to promote stability on the roof.

The addition of these fillers, however, does not improve the relatively deficient waterproofing characteristic of the bitumen and reduces the percentage of bitumen.

These materials, while less susceptible to slide at higher temperatures, have a tendency to absorb moisture, oxidize, crack, disintegrate, and develop weaknesses which result in unsightly "cheesiness" or serious leaks. These weaknesses are acelerated where no wearing surface is provided. The period of serviceability depends upon (1) the amount of protection given to a bituminous material, and (2) the quality of the bituminous material. When oxidation, cracking and disintegration have progressed to the stage permitting water to reach the underlying fabric, disintegration of the fabric sets in and the usefulness of the roof is practically at an end.

The more completely the bituminous material, or membrane, is protected by slag, gravel, tile, etc., the slower the rate of change in the material. Where the bitumen is unprotected, obviously the rate of change is rapid and the life of the roof materially curtailed. Non-technically, asphalts in general fall short of giving ideal protection against water and climatic change for extended periods of time comparable with the life of other building elements.

It is an object of the present invention to provide new tar products which are relatively free of the disadvantages of ordinary tar products or asphalt products.

A further object is to provide products having properties which render them easy to apply, resistant to weathering, free from tendencies to bleed at the highest temperatures and from brittleness at the lowest temperatures of normal use, water repellent, and unaffected by fermentation or decay.

Further objects will appear from the following description of my invention.

It is known that the character of coal-tar pitch may be modified by heating the pitch with coal, such as bituminous coal, cannel coal, lignite, and the like. Such products are shown, for example, in Austrian Patent 90,380, British Patent 316,897, and United States Reissue Patent 17,762. The products of the present invention differ from such prior products in that they possess a combination of properties adapting them for application as roofing pitches to steep roofs. While tar products have been applied to roofs with a slope perhaps as steep as three inches per horizontal foot, they could not be employed satisfactorily upon steeper roofs. The products of the present invention, on the other hand, are adapted for application to roofs having a slope as steep as five or six or more inches per horizontal foot. They are similarly adapted for use in conjunction with felt or fabric in waterproofing other steep construction surfaces than roofs.

The products of the present invention are characterized by a softening point between about 50° C. and about 90° C.; a penetration at 0° C. of at least 4, and a penetration at 25° C. not more than five times the penetration at 0° C. They are further characterized by a rubbery feel which distinguishes them from ordinary coal-tar

pitches. Moreover, they retain all the desirable properties of ordinary coal-tar pitch, such as water repellency, oxidation resistance, and toxicity towards microorganisms of decay. In addition, they have pronounced non-sliding characteristics at the highest temperatures of service and are less brittle and liable to crack at the lowest temperatures of service. In these respects, they are equal to or superior to roofing asphalts.

The products of the present invention may be obtained by heating a mixture of tar oil and pitch constituents with coal under certain specific conditions of control to be hereinafter more fully delineated.

The mixture of tar oil and pitch constituents normally should have a specific gravity between 1.1 and 1.22 at 38° C., compared with water at 15.5° C., and preferably between 1.10 and 1.20. It should have an "Engler" specific viscosity not less than 2.0 at 80° C. It should contain not more than 5% of oils distilling off below 235° C. and not more than 25% of oils distilling off below 300° C., when tested according to A. S. T. M. Standard Method D20–30. The residue of distillation to 300° C. should have a float test not above 130 seconds and normally not above 80 seconds at 70° C. Such a mixture may be prepared by mixing with a coal tar an added quantity of high-boiling coal tar oils; the coal tar may be previously distilled to remove constituents boiling below about 235° C. or the mixture may be distilled for this purpose. In place of coal tar and coal-tar oil, water gas tar and water-gas-tar oil, respectively, may be employed.

The tar-oil-pitch-constituent mixture above defined is not an ordinary coal tar or coal-tar pitch and is distinguished from such products by its high content of high-boiling oils which results in the low float test of distillation residue previously indicated. Coal-tar pitches of equally low low-boiling distillate content ordinarily have a distillation residue float test of 150 to 300 seconds at 70° C. It is distinguished from ordinary tar oils on the other hand by its content of pitch constituents, reflected in its high viscosity.

The products of my invention may best be made by employing a good grade of bituminous coal. Anthracite has not been found suitable. Sub-bituminous coals, lignite and brown coal, for example, are dispersable only with difficulty. My experience has indicated that, to avoid "gel-like" products hereinafter discussed, higher digestion temperatures are required for these coals and, since dispersion is less complete, correspondingly more coal should be used, and undispersed residue separated. Several cannel coals have been employed with some success. The percentage of coal digested in the digestion medium may be between 7% and 25% and preferably between 15% and 25% of the total mixture; this refers to the percentage of coal actually dispersed in the tar oil pitch constituent mixture and does not include ash or other solid matter which may be removed by sedimentation from the hot digested material. Coals classified as bituminous according to rank are the most satisfactory, since with all of such coals that I have employed homogeneous products having the desired properties are readily obtained. Among the bituminous coals, I have found that those having pronounced coking properties are very suitable for digestion. Coals of low volatile content are especially desirable, since their digestion is more easily controlled, and superior products are obtained. Among the coals which I have found satisfactory for preparation of products of my invention are lower Freeport Seam steam coal, Pennsylvania low-volatile bituminous coal, Ohio non-coking coal, low-volatile West Virginia coking coal, and German bituminous coals (Steinkohlen).

The heating process for producing the products of the present invention must be carefully controlled. For the digestion of any one coal to produce a maximum dispersion of coal constituents a certain minimum temperature of heating is required. This temperature produces gel-like products which are relatively unstable at high temperatures and of unsuitable physical properties for application to the roof. Furthermore, such gel products are difficult to control in production. The minimum temperature referred to is inadequate to produce the products of my invention and I have found that a temperature between 20° and 100° higher and preferably between 30° and 70° higher should be employed.

A suitable test to establish the digestion temperature required for making the products of the present invention involves heating the coal under consideration with a suitable digestion medium for a fixed period of time at a series of digestion temperatures and determining the consistency of the products thus obtained. The product having the maximum softening point will have been digested at a temperature lower than suitable for the purposes of my invention. In making such a series of determinations, consecutive temperatures should be selected sufficiently close so that the temperature of maximum consistency or maximum softening point will be reasonably close to one of the temperatures actually selected. The following procedure has been found to be highly satisfactory.

The digestion medium comprises a high-boiling coal-tar oil having the following characteristics:

Specific gravity, 38° C./15.5° C	1.177
Distillation (A. S. T. M. Standard Method D246–33):	
Distillate to 210° C. per cent	0
Distillate to 235° C. do	0
Distillate to 270° C. do	0
Distillate to 315° C. do	0
Distillate to 355° C. do	10.0
Float test at 70° C. on distillation residue seconds	33

To 75 parts of this oil 25 parts by weight of the pulverized coal to be tested are added. The mixture is heated with agitation for five hours at a temperature previously selected, for example 245° C., and the softening point of the product is determined every hour. Another sample is then heated at a higher temperature, say 270° C., for a similar period, and third and fourth samples may be heated at 295° and 320° C. for a similar period, the softening points of the products in each case being determined at hourly intervals after the selected temperature is reached.

From a series of such determinations it will be found that the softening point rises with digestion temperature and time until it reaches a maximum. This may be explained on the basis that as the temperature rises, more and more coal becomes dispersed until finally all of the constituents capable of dispersion are dispersed. As the temperature rises further, decomposition of the dispersed constituents takes place, as evidenced by evolution of fixed gases and volatile liqueflable hydrocarbons containing paraffinic bodies, the formation of acidic constituents, and a change in

the proximate analysis of the digested coal, evidenced by a reduction in volatile combustible matter. The softening points of the products obtained at higher digestion temperatures fall below the maximum. The type of oil described is a particularly suitable digestion medium for the purpose since its use permits ready discernment of the maximum consistency. The large percentage of coal selected also favors observation of the changes in properties produced by changes in the coal.

The preferred products of the present invention exhibit the following characteristics:

Softening point : between 60° and 80° C.
Penetration at 0° : at least 6
Penetration at 25° C. : between 20 and 40
Penetration at 46.1° C. : not more than 120
Per cent soluble in carbon disulfide : at least 20
Specific gravity 25° C./25° C. : at least 1.20

For commercial operations and particularly for manufacture of products of the preferred type it is desirable that the optimum temperature for digesting the coal be determined. For this purpose a second set of tests may be run at less widely separated temperatures and with a digestion medium more similar to that used for preparing the products. Thus I have found a mixture consisting of 50% by weight of a high-boiling, coal-tar oil and 50% of a topped coke oven tar to be a highly satisfactory digestion medium, and a temperature interval of about 10° C. sufficiently narrow for my purposes. A typical coal-tar oil, considered as characteristic of a desirable oil component of the digestion medium, has the following test properties:

Specific gravity, 38° C./15.5° C	1.142
Distillation (A. S. T. M. Standard Method D246–33):	
Distillate to 210° C, per cent	0
Distillate to 235° C, do	0
Distillate to 270° C, do	1.8
Distillate to 300° C, do	6.5
Distillate to 315° C, do	10.5
Distillate to 355° C, do	32.3
Float test at 70° C. on distillation residue, seconds	31

The topped coke-oven tar, and the mixture (equal parts of oil and tar) comprising the digestion medium have the following characteristics:

	Tar	Mixture
Specific gravity at 38° C./15.5° C		1.174
Specific gravity at 25° C./25° C	1.222	
Distillation (A. S. T. M. Standard Method D20–30):		
Distillate to 170° C, percent	0	0
Distillate to 235° C, do	1.6	0.7
Distillate to 270° C, do	7.2	4.0
Distillate to 300° C, do	14.4	11.2
Float test at 70° C. of distillation residue, seconds		26
Softening point of distillation residue, ° C	53	

The second series of digestions is conducted with standardized conditions and a heating period of about one hour at the digestion temperature. For these tests sufficient coal is employed to yield products having a softening point of some definite value; for instance about 93° C. or about 68° C. The 93° C. softening point is advantageous since it has been found that this softening point may be obtained with relatively minor variations from 25% coal employed in the tests previously considered. The products of this series of tests are further tested for their penetration at 0° C., 25° C., and 46.1° C. and are distilled by method D20–30 of the American Society for Testing Materials, and the softening points of the distillation residues are determined. If a product during this distillation tends to foam excessively, the digestion temperature employed was below the optimum temperature. This test serves as a clarification of the softening point tests of the first series of runs. Of course the product should be homogeneous. When reheated to liquefaction temperature it should liquefy without prolonged heating.

A comparison between the softening point of the product and that of the residue remaining after the distillation test may be used to establish the stability of the product towards heat. Thus, if the softening point of the distillation residue is lower than that of the product before distillation, or is not higher in proportion to the percentage of distillate, as compared to changes in softening point shown by heat-stable pitches under similar distillation conditions, the product is undesirably lacking in stability and a higher digestion temperature should be employed; on the other hand, if the softening point increases in a normal manner as a result of the distillation test, and the increase is proportional to the percentage of distillate, it may be concluded that the digestion temperature is adequately high to produce a stable product. For example, a heat-stable pitch of the preferred properties will ordinarily show an increase in softening point on distillation of at least 1° C. for each percent of distillate to 300° C. obtained in the distillation test.

It is advantageous to employ as low a digestion temperature as permissible while securing the desired results since heating on a commercial scale to higher temperatures requires more heat, which serves no useful function. Moreover, further increases in temperature result in an increase in the temperature susceptibility of the product; and since my preferred products possess a very low temperature susceptibility, they will not be obtained if the digestion temperature is excessive.

The following results with six American bituminous coals selected at random from the results of an examination of a large number of coals, demonstrate the results that may be expected in application of the tests previously described.

Coal No.	1	2	3	4	5	6
Digestion in high-boiling coal tar oil:						
Observed dig. temp. for max. consistency °C., degrees	245	245	270	270	295	295
Approx. max. consistency, softening point, °C., degrees	93	85	116	93	154	143
Digestion in tar, oil mixture:						
Selected optimum digestion temperature °C., degrees	305	305	315	305	345	335
Coal req. for 93° C. softening point, per cent	26	27.5	23	25	20	17

It may be observed that the difference for these coals between the digestion temperatures for maximum consistency and the optimum digestion temperatures ranges between 35° C. and 60° C. Once the optimum digestion temperature has been determined for any particular coal, that temperature may be employed without variation for the preparation of any number of batches of products from that coal. Digestion

at temperatures substantially higher than the optimum will result in increasing the temperature-susceptibility of the product and usually in a product of lower penetration at 25° C. With certain coals it may be advantageous to employ a digestion temperature somewhat higher than the optimum for the purpose of modifying the character of product within a desired range.

It will be evident that for the preparation of the preferred products of my invention the proportion of coal employed should be somewhat below the 25% employed in the tests to determine the digestion temperature of maximum consistency and also below the percentage employed in the second group of tests listed above where amounts of coal from 17% to 27½% of the digestion mixture were used. The exact amount of coal for the production of a particular product will depend upon the properties of the digestion medium employed and the digestion characteristics of the coal used. The digestion media suitable for preparation of products of the present invention have previously been defined. However, within the definition given, considerable latitude may be exercised and the question of proportions within the indicated limits is a matter of intelligent selection of the most economical material available and proportions may be varied to suit prevailing economic conditions. The nature of the changes resulting from varying percentages of constituents cannot be strictly generalized but it may be said that with use of a coke-oven tar and a high-boiling oil having the specific characteristics of the products employed as a digestion medium in the temperature selection tests above described, the addition of 1 per cent of coal and subtraction of 1 per cent of tar from the formula will usually increase the softening point of the product between about 4° and about 9° C.

In general products of the present invention are made by heating together the coal with a digestion medium having the properties previously described to the digestion temperature. It is not necessary that digestion be continued any particular length of time after the digestion temperature has been attained, nor does heating for a reasonable length of time at this temperature detrimentally affect the product.

In the treatment of bituminous coal in which substantially all of the combustible constituents occur in the product as dispersable decomposition products, part of the ash constituents may be permitted to settle out or all may remain in the final product. In practice, if the coal has been digested in lump form, it is desirable to settle out adventitious material such as slate which has not been disintegrated by the digestion. With this procedure normal coal ash remains dispersed to the extent of, say 60% to 100%. If the coal was digested in powdered form, disintegration is more complete and only a small part of the ash can be settled out unless excessively long settling periods are used. Fusain in general remains completely dispersed.

The digestion process may be carried out in any suitable apparatus equipped with adequate heating means and an agitating device. Normally a condenser should be employed for condensing oil vapors evolved during the heating process. These oils may be separately collected. The charge is gradually heated with continued agitation to the desired digestion temperature. The charge is then allowed to cool, preferably with continued agitation, until the temperature has dropped to a point where the product can be conveniently withdrawn in liquid condition. Minor changes in the softening point of the product may be made as required by adding additional tar, tar oil, or pitch to the product prior to its solidification.

The heating period in practice will depend upon the quantity of material in the heating vessel, the efficiency of the stirring apparatus, the amount of heating surface, etc. In batch operations the period of heating to raise the temperature to around 300° C., may be only a couple of hours or less for small quantities of material, up to eight or ten or more hours with larger amounts, especially where agitation is less rapid. The principal consideration in determining the rate of heating is to avoid overheating of portions of the charge.

The apparatus employed for making the products of my invention may suitably comprise an ordinary, direct-fired still, for example a horizontal cylindrical still constructed of steel. A mechanical agitating device advantageously may be provided within the vessel adapted to prevent local overheating and to assure uniform conditions of treatment.

The following examples illustrate the invention.

Example 1.—A bituminous coal obtained from the Lower Freeport Seam in Pennsylvania was pulverized to pass a 10 mesh sieve. A representative sample of this coal was ground to pass a 60 mesh sieve; it had the following proximate analysis:

	Per cent
Moisture	1.10
Volatile matter	29.37
Fixed carbon	59.85
Ash	9.68

Four samples of 25 parts by weight of the pulverized coal passing a 10 mesh sieve were added to four samples of 75 parts by weight of a high-boiling coal-tar oil having a specific gravity at 38° C./15.5° C. of 1.177 and 10% of distillate to 355° C. by A. S. T. M. Standard Method D246–33. The several mixtures were heated while stirring to temperatures of 245° C., 270° C., 295° C., and 320° C., respectively, and maintained at these temperatures for a period of five hours. During this period thief samples were taken hourly and softening point tests were made on these samples. Results of these tests were as follows and may be regarded as accurate within a range of about plus or minus 2° C.

Digestion temperature	Period of digestion, hrs.	0	1	2	3	4	5
		°C.	°C.	°C.	°C.	°C.	°C.
245° C.	Softening point	88	94½	93	94	93	93
270° C.	Softening point	95	93	96	92	93	89½
295° C.	Softening point	91	87	85	82	82	82
320° C.	Softening point	82	76	77	75	72	75½

From these tests it will be observed that the lowest digestion temperature of maximum softening point for this coal is in the neighborhood of 245° C. The optimum digestion temperature which lies between 20° and 100° C. above was then determined as follows:

A mixture was prepared consisting of 50 parts of the high-boiling coal-tar oil having the following properties: specific gravity at 38° C./15.5° C. 1.142; 1.8% of distillate to 270° C., 6.5% to 300° C., 10.5% to 315° C., and 32.3% to 355° C. by

A. S. T. M. Standard Method D246–33; and 50 parts of a topped coke oven tar having the following properties: specific gravity at 25° C./25° C. 1.222; 1.6% of distillate to 235° C., 7.2% to 270° C., and 14.4% to 300° C. by A. S. T. M. Standard Method D20–30. To 75 parts by weight of this mixture 25 parts by weight of pulverized coal were added and the mixture was heated with agitation in a period of about two and one-half hours to a temperature of about 290° C. and was maintained at this temperature for one hour. The product was cooled to about 200° C. with agitation, after which it was allowed to solidify. The product was then carefully melted, tested for softening point, and distilled to a temperature of 300° C. by A. S. T. M. Standard Method D20–30. During the distillation excessive foaming was observed.

Two additional tests were made with the same digestion medium and the same coal, employing digestion temperatures of 300° C. and 310° C., respectively. In order to obtain products of about the same softening point as obtained by the digestion at 290° C., somewhat higher percentages of coal were digested at the higher temperatures. For these two tests the digestion procedure was the same as for the tests at 290° C. and the products were tested in a similar manner. The results of the tests were as follows:

Digestion temperature ° C	290	300	310
Digestion medium parts by weight	75	74.5	74
Coal do	25	25.5	26
Softening point of product ° C	94½	92	93
Distillate to 300° C	9.5	9.1	8.9
Softening point of distillation residue ° C	99	100½	104

The products of digestion at 300° C. and 310° C. were homogeneous and liquefied readily on heating. The product of digestion at 300° C. foamed slightly on distillation and the distillation resulted in a change in softening point of 0.9° C. for each percent of distillate to 300° C. The product of digestion at 310° C. did not foam excessively on distillation and the distillation resulted in a change of softening point of 1.2° C. for each percent of distillate to 300° C. The tests thus established an optimum digestion temperature of about 300° C. to 310° C.

51 parts of dehydrated coke oven tar and 30 parts of heavy coal tar distillate were then mixed to provide a digestion medium having properties within the range previously described and 19 parts of pulverized bituminous coal from the Lower Freeport Seam, Pennsylvania were added. The mixture was then heated to and maintained for one hour at a temperature of about 310° C. During the digestion the mixture was agitated and oil vapors were withdrawn. The oil thus removed and condensed amounted to 7.7% by volume of the digestion medium. The product recovered had the following properties:

Softening point ° C	68
Penetration at 0° C	17
Penetration at 25° C	32
Penetration at 46.1° C	96

Example 2.—18,671 pounds of bituminous coal from the Lower Freeport Seam, Pennsylvania were pulverized to pass a No. 8 sieve. The coal was charged in a commercial still equipped for agitation and containing 38,770 pounds of dehydrated coke oven tar and 28,158 pounds of high-boiling coal tar oil. The mixture of coke-oven tar and coal tar oil had properties within the range previously disclosed. The charge was heated to about 310° C. in about eight hours, and maintained at that temperature for about one hour, removing volatile oils. After settling for one hour, the product was withdrawn from the still, and had the following properties:

Softening point ° C	70
Penetration at °0 C	14
Penetration at 25° C	30
Penetration at 46.1° C	80
Specific gravity 25° C./25° C	1.227
Insoluble in carbon disulfide per cent	28
Ash do	1.24
Distillation, A. S. T. M. Standard Method D20–30 distillate to 300° C do	8.8
Softening point of distillation residue ° C	87

Example 3.—A product prepared from a liquid digestion medium meeting the above described requirements and comprising 50 parts of dehydrated coke oven tar and 25 parts of heavy coal-tar distillate, and 25 parts of coal, by digesting at 300° C. with the removal of 3.3% of volatile oils, had the following characteristics:

Softening point ° C	80
Penetration at 0° C	5
Penetration at 25° C	14
Penetration at 46.1° C	37

Example 4.—To a tar:tar-oil mixture having properties within the range above described and prepared from 72 parts of dehydrated coke oven tar and 10 parts of heavy water gas tar distillate, 18 parts of pulverized coal were added. The mixture was heated to 320° C. Volatile oils amounting to 4.4% by volume of the tar and distillate were distilled off during the heating period. The product was then allowed to cool. The product possessed the following characteristics:

Softening point ° C	82
Penetration at 0° C	22
Penetration at 25° C	40
Penetration at 46.1° C	76

Example 5.—To a mixture of 42.5 parts by weight of high-boiling coal-tar oil and 42.5 parts by weight of dehydrated coke-oven tar having the following properties:

Specific gravity 35° C./15.5° C	1.155
Specific viscosity, Engler, at 80° C	2.3
Distillation, A. S. T. M. Standard Method D20–30:	
Distillate to 170° C per cent	0
Distillate to 235° C do	3.5
Distillate to 270° C do	11.3
Distillate to 300° C do	20.2
Float test at 70° C. on distillation residue seconds	29

15 parts by weight of low-volatile bituminous coal were added. The mixture was digested at a temperature of 340° C. for one hour, removing volatile oils amounting to 12.5% by volume of the digestion medium. The product had the following properties:

Softening point ° C	70
Penetration at 0° C	26
Penetration at 25° C	69
Penetration at 46.1° C	193

The tests outlined throughout this specification are performed in accordance with standard apparatus and procedure prescribed by the American Society for Testing Materials. Softening points are determined by the ring and ball method. Penetration is given in units of one-

tenth of a millimeter with weights and periods as follows:

At 0° C.	200 grams, 60 seconds
At 25° C.	100 grams, 5 seconds
At 46.1° C.	50 grams, 5 seconds

This application is a continuation in part of my copending application Serial No. 749,618, filed October 23, 1934.

I claim:

1. A heat-stable bituminous product, particularly adapted for application by remelting, obtainable by heating a digestion medium, comprising tar oil and pitch constituents and having a float test of residue from distillation to 300° C. not above 130 seconds at 70° C. and containing distillable oils including not more than 5% distillable below 235° C. and not more than 25% distillable below 300° C., with between 7% and 25% of a coal of the group consisting of bituminous coal and sub-bituminous coal, the perecentage being based upon the total mixture of digestion medium and coal, to a temperature between 20° and 100° above the digestion temperature corresponding to a product of maximum softening point, said bituminous product having a softening point between about 50° and about 90° C., a penetration at 0° C. at least 4, and a penetration at 25° C. not more than five times the penetration at 0° C.

2. A heat-stable bituminous product suitable for use as a steep-roofing pitch, particularly adapted for application by remelting, and obtainable by heating a digestion medium, comprising tar oil and pitch constituents and having at 38° C./15.5° C. a specific gravity between 1.1 and 1.22, an Engler specific viscosity not less than 2.0 at 80° C., a float test of residue from distillation to 300° C. not above 130 seconds at 70° C. and containing distillable oils including not more than 5% distillable below 235° C. and not more than 25% distillable below 300° C., with between 7% and 25% of bituminous coal, the percentage being based upon the total mixture of digestion medium and coal, to a temperature between 20° and 100° above the digestion temperature corresponding to a product of maximum softening point, said bituminous product having a softening point between about 50° and about 90° C., a penetration at 0° C. at least 4, and a penetration at 25° C. not more than five times the penetration at 0° C.

3. A heat-stable bituminous product suitable as a coating and bonding material for coal-tar-pitch-impregnated roofing fabric on steep roofs, and particularly adapted for application by remelting, said bituminous product being obtainable by heating a digestion medium, comprising tar oil and pitch constituents and having at 38° C./15.5° C. a specific gravity between 1.10 and 1.20, an Engler specific viscosity not less than 2.0 at 80° C., a float test of residue from distillation to 300° C. not above 80 seconds at 70° C. and containing distillable oils including not more than 5% distillable below 235° C. and not more than 25% distillable below 300° C. with between 15% and 25% of bituminous coal, the percentage being based upon the total mixture of digestion medium and coal, to a temperature between 20° and 100° above the digestion temperature corresponding to a product of maximum softening point, said bituminous product having a softening point between about 60° C. and about 80° C., a penetration at 0° C. at least 6, a penetration at 25° C. between 20 and 40, and a penetration at 46.1° C. not more than 120.

4. The method of preparing a bituminous product, which comprises heating a digestion medium, comprising tar oil and pitch constituents and having a float test of residue from distillation to 300° C. not above 130 seconds at 70° C. and containing distillable oils including not more than 5% distillable below 235° C. and not more than 25% distillable below 300° C. with between 7% and 25% of a coal of the group consisting of bituminous coal and sub-bituminous coal, the percentage being based upon the total mixture of digestion medium and coal, to a temperature between 20° and 100° above the digestion temperature corresponding to a product of maximum softening point.

5. The method of preparing a bituminous product suitable for use as a steep-roofing pitch, which comprises heating a digestion medium comprising tar oil and pitch constituents and having at 38° C./15.5° C. a specific gravity between 1.1 and 1.22, an Engler specific viscosity not less than 2.0 at 80° C., a float test of residue from distillation to 300° C. not above 130 seconds at 70° C. and containing distillable oils including not more than 5% distillable below 235° C. and not more than 25% distillable below 300° C., with between 7% and 25% of bituminous coal, the percentage being based upon the total mixture of digestion medium and coal, to a temperature between 20° and 100° above the digestion temperature corresponding to a product of maximum softening point.

6. The method of preparing a bituminous product suitable as a coating and bonding material for coal-tar-pitch impregnated roofing fabric on steep roofs, which comprises heating a digestion medium, comprising tar oil and pitch constituents and having at 38° C./15.5° C. a specific gravity between 1.10 and 1.20, an Engler specific viscosity not less than 2.0 at 80° C., a float test of residue from distillation to 300° C. not above 80 seconds at 70° C. and containing distillable oils including not more than 5% distillable below 235° C. and not more than 25% distillable below 300° C., with between 15% and 25% bituminous coal, the percentage being based upon the total mixture of digestion medium and coal, to a temperature between 20° and 100° above the digestion temperature corresponding to a product of maximum softening point.

BENJAMIN ALBERT ANDERTON.