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[54] **MANUFACTURE OF LIGNITE BINDER PITCH**

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[51] **Int. Cl.** ... **C08h 13/00,** C08h 17/08, C10c 3/00

[58] **Field of Search**..... 106/38.8, 273, 284; 208/22, 45; 264/29, 105

[56] **References Cited**

UNITED STATES PATENTS

2,683,107	7/1954	Juel	106/284 X
2,955,079	10/1960	Foley	208/22

FOREIGN PATENTS OR APPLICATIONS

823,381	11/1959	Great Britain	106/284
828,116	2/1960	Great Britain	106/284

OTHER PUBLICATIONS

Abraham, Asphalts and Alied Substances, 6th Ed., Vol. two, N.Y. D. Van Nostrand Co. Inc., TN 853, A35 1960 C.4. pages 39–51 relied on.

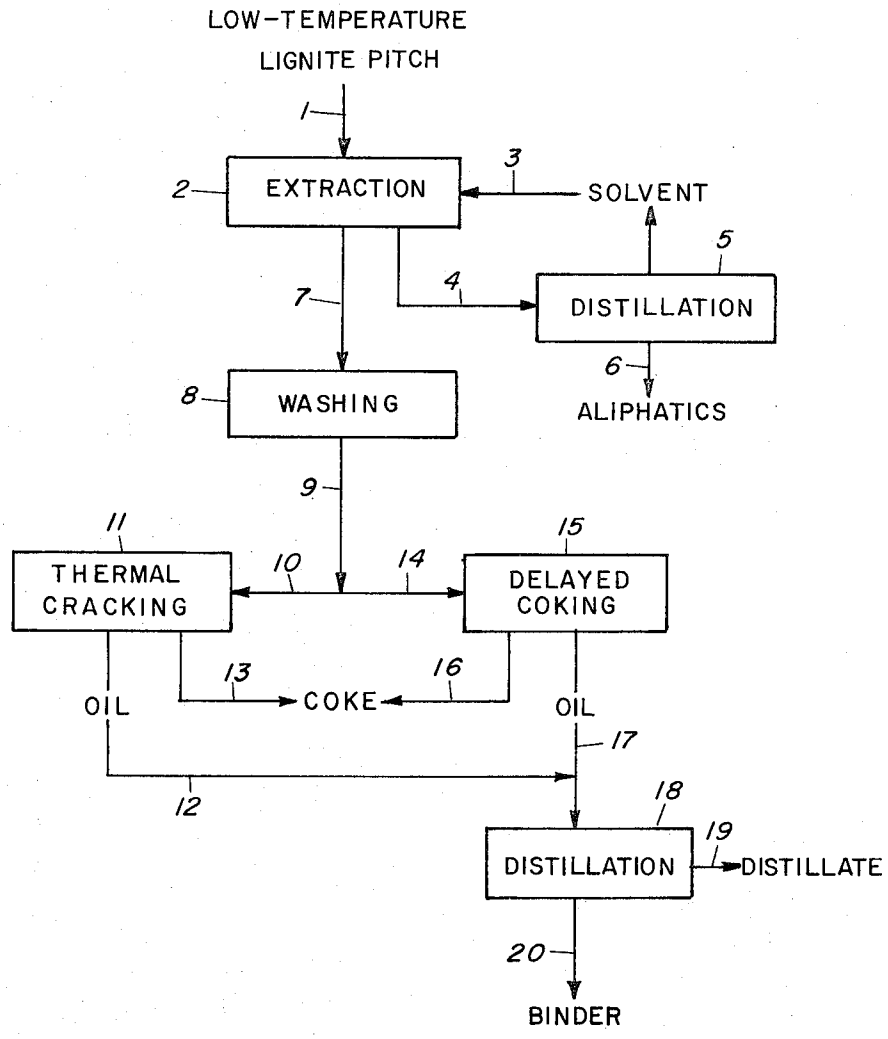
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[57] **ABSTRACT**

Binder pitch suitable for metallurgical electrode production is prepared from crude pitch obtained from the low-temperature carbonization of lignite. Aliphatic hydrocarbons contained in the crude pitch are removed by solvent extraction prior to thermal treatment such as delayed coking or thermal cracking. Binder pitch product is recovered as a residual fraction of the oil produced in the thermal treatment.

6 Claims, 1 Drawing Figure



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MANUFACTURE OF LIGNITE BINDER PITCH

BACKGROUND OF THE INVENTION

Low temperature carbonization of lignite produces tar and a char residue. The char finds use as power plant fuel, for synthesis gas production and for blending with other coals to produce metallurgical coke. Low temperature tar can be utilized as a source material for organic chemicals and binder pitches for use in making carbon electrodes.

Pitch comprises the residue from crude tar distillation. It typically has a boiling point above about 350°C and is an amorphous solid material at room temperature. Lignite pitch differs from pitch derived from bituminous coal tars in that it generally has a lower percentage of fixed carbon and a lower carbon/hydrogen atomic ratio.

We have previously found that lignite pitch can be upgraded by either delayed coking techniques or by thermal cracking to produce a number of useful products. When the pitch is thermally cracked, products include coke, oil and a gas rich in ethylene. Distillation of the oil fraction leaves a pitch residue having desirable characteristics for use as a binder material. Electrodes prepared from this binder pitch and lignite coke showed somewhat higher carbon consumption, higher resistivity and lower physical strength than did standard electrodes when used in alumina reduction.

SUMMARY OF THE INVENTION

We have now found that solvent extraction of the aliphatic hydrocarbons contained in low temperature lignite pitch substantially improves the quality of binder pitch recovered as a product of delayed coking or thermal cracking. Preferred solvents include saturated, aliphatic hydrocarbons having about 7 to 9 carbon atoms.

Hence, it is an object of this invention to provide an improved method for processing low temperature lignite pitch.

Another object of this invention is to recover aliphatic hydrocarbons from lignite pitch.

A specific object of this invention is to produce an improved binder material from lignite pitch.

DETAILED DESCRIPTION OF THE INVENTION

Our invention will best be understood and appreciated from the following description of the process taken in connection with the accompanying drawing in which:

The FIGURE is a diagrammatic flow sheet of our process.

Referring now to the drawing, a low temperature lignite pitch material **1**, preferably in the form of pulverized particles, is fed either intermittently or continuously into extraction zone **2**. There, the pitch material is contacted with a solvent selective for aliphatic hydrocarbons introduced by way of line **3**. Extraction may be accomplished using a variety of conventional techniques and apparatus on either a batch or continuous basis. Generally it is preferred to maintain an excess of solvent within the extraction zone; a weight ratio of solvent to pitch ranging from about 2:1 to 3:1 giving satisfactory results. Extraction temperatures preferably range from room temperature to about the boiling point of the solvent or from about 20° to about 100°C. However, even higher temperatures may be used. Contact time ranges from a few minutes to about 2 hours depending upon temperature and solvent to pitch ratios.

In the extraction zone, aliphatic hydrocarbon components of the pitch are leached from the pitch particles and are dissolved in the solvent. A solvent fraction, rich in dissolved aliphatics, is separated from the pitch particles by settling or filtration, and is passed via line **4** to distillation zone **5.** A bottoms fraction **6** comprising extracted aliphatic hydrocarbons is recovered as a product of the process while the overhead stream comprising solvent is routed back to extraction zone **2.**

Leached pitch particles are passed by means **7** to washing zone **8.** There, the pitch is washed with a small quantity of solvent which may be recovered by distillation. Pitch particles now substantially depleted in aliphatic hydrocarbon content, are then subjected to a high temperature processing step which may comprise either thermal cracking or delayed coking. If thermal cracking is utilized, then pitch is passed from washing zone **8** to thermal cracking zone **11** by way of conduit means **9** and **10.** Thermal cracking may be carried out in conventional fashion at temperatures generally in the range of 700° to 900°C to yield an oil fraction **12,** a coke residue **13** and of course a gas stream (not shown).

If delayed coking techniques are utilized, then the pitch is passed to delayed coking zone **15** by way of conduit means **9** and **14.** Like in thermal cracking, products of the delayed coking step comprise a coke residue **16** and an oil fraction **17.** The delayed coking step is carried out in conventional fashion in the same manner used in the delayed coking of petroleum residuals. Oil obtained from either thermal treatment is then passed to distillation zone **18** where it is separated into an overhead distillate stream **19** and a residual pitch binder fraction **20.** Distillate stream **19** finds use as a raw material for the separation or synthesis of a variety of aromatic compounds while the pitch fraction constitutes a high quality binder for electrodes, pitch fiber pipe and roofing materials.

Properties and composition of low temperature lignite pitch used as the starting material in our process will vary somewhat depending upon the properties of the lignite and the conditions used in the low temperature carbonization step. A typical analysis of the material is as follows:

TABLE 1

Ultimate Analysis:	Percent
Carbon	84.7
Hydrogen	8.5
Nitrogen	0.9
Oxygen	4.6
Chlorine	0.01
Sulfur	0.9
Moisture	0.00
Flash Point (°F)	510
Softening Point (R & B) glycerin (°C)	90
Specific Gravity (25 C/25 C)	1.128
Bitumen, soluble in CS_2 (%)	78.8
Conradson carbon (%)	20.8

In addition, extractable aliphatic content of the low temperature pitch ranges from about 5 to about 20 percent and commonly is on the order of 8–10 percent. Bituminous pitch, on the other hand, contains only traces of aliphatics.

TABLE 3

Binder (%)	Electrode Strength (Kg/cm²) Aliphatics Present	Aliphatics Removed	Electrode Resistivity (ohm-cm) Aliphatics Present	Aliphatics Removed
25	240	300	0.009	0.010
28	201	241	.013	.018
29	205	256	.013	.012
30	208	237	.012	.011
30	213	321	.013	.011

Solvents suitable for use in the extraction step generally comprise saturated, paraffinic hydrocarbons of intermediate boiling range. Preferred solvents include paraffinic hydrocarbons having 7 to 9 carbon atoms. It was found that n-heptane was suitable for use but that n-hexane and n-pentane were less satisfactory in that additional components of the pitch in addition to the aliphatic fraction were extracted. Our most preferred solvents comprise branched chain paraffinic hydrocarbons having 8 carbon atoms. Trimethylpentane is particularly preferred. Of course, mixtures of these preferred solvents are in most cases equally effective.

While any reduction in aliphatic content of the crude pitch tends to improve quality of the product binder pitch, we prefer to extract more than 75 percent and most preferably more than 90 percent of the aliphatics contained in the crude pitch. Aliphatics extracted generally have a boiling point in excess of about 300°C and find use in the production of biodegradable synthetic detergents.

The following example illustrates a specific embodiment of our process.

EXAMPLE

A crude lignite pitch, having properties as set out in Table 1, was obtained by low temperature carbonization of lignite coal. It was processed in accordance with the flowsheet set out in the FIGURE using trimethylpentane as a solvent in the extraction step. After thermal cracking and distillation, the binder pitch product obtained had the following characteristics:

TABLE 2

Carbon (%)	89.9
Hydrogen (%)	5.03
Carbon/hydrogen atomic ratio	1.49
Softening point (°C)	113

A second binder pitch was prepared in a similar fashion except that the aliphatic extraction step was omitted. The two pitches were then evaluated for use as a binder material for making carbon electrodes for metallurgical use. Results of these comparative tests are as follows:

As may be seen from Table 3, removal of aliphatics from the crude pitch results in improved properties of the binder pitch. Electrodes prepared using a binder pitch from which aliphatics had been removed showed generally lower electrical resistivity and substantially higher physical strength than did electrodes prepared from aliphatic-containing pitch.

What is claimed is:

1. A process for producing a composition suitable for use as a binder for carbon particles which comprises:

treating a crude pitch, obtained as the distillation residue of tar produced by the low temperature carbonization of lignite coal and further characterized by containing from about 5 to 20 percent extractable aliphatic hydrocarbons, by solvent extraction, using as a solvent a liquid consisting essentially of a saturated aliphatic hydrocarbon having 7 to 9 carbon atoms, to remove a substantial portion of aliphatic hydrocarbons contained in said crude pitch fraction;

thermally cracking the residue from said solvent extraction step at temperatures within the range of 700° to 900°C to produce an oil fraction a gas fraction and a coke fraction, and

distilling said oil fraction to recover an overhead distillate stream comprising aromatic compounds and a residual binder pitch product.

2. The process of claim **1** wherein more than 75 percent of the aliphatic hydrocarbons contained in said crude pitch are removed in the solvent extraction step.

3. The process of claim **2** wherein rich solvent from the extraction step is distilled to recover an aliphatic hydrocarbon product stream and a recycle solvent stream.

4. The process of claim **3** wherein the solvent is a branched chain aliphatic hydrocarbon having 8 carbon atoms.

5. The process of claim **4** wherein the solvent is trimethylpentane.

6. The process of claim **3** wherein said pitch binder product is mixed with particulate coke and is formed into an electrode.

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