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(54) Process for producing premium coke.

(57) A premium coke is made by the delayed coking of a blend of pyrolysis tar and hydrotreated decant oil. This premium coke is used for the production of graphite electrodes having a low coefficient of thermal expansion.

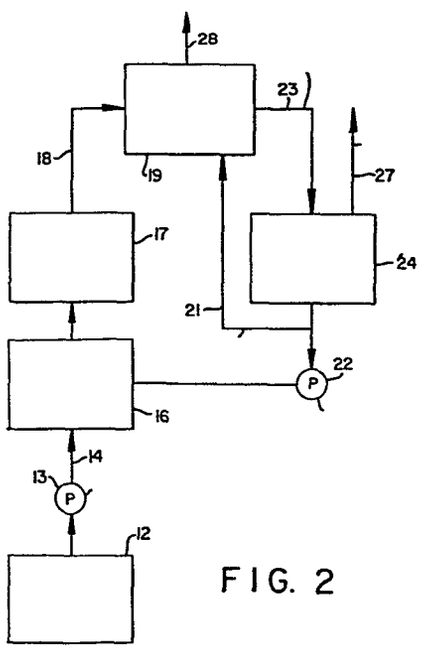

FIG. 2

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Process for producing a premium coke suitable for use in the production of a graphite electrode and graphite electrode made from said premium coke.

The invention relates to premium coke suitable for use in the production of a graphite electrode, and particularly to a process for producing a premium coke from a blend of pyrolysis tar and hydrotreated decant oil.

Premium coke is well known in the art and is a commercial grade of coke having acicular, anisotropic microstructure.

The premium cokes are used in the production of electrode grade graphite. This use of premium cokes results in various requirements to be made of the cokes. Some of these requirements are pointed out herein.

A graphite electrode which will be used in the arc melting of steel or the like must possess a low value for the coefficient of thermal expansion (CTE) because of the severe thermal shocks which occur in such processes. The premium coke used for producing the graphite electrode must be capable of imparting a low CTE to the electrode.

The process for producing a graphite electrode from a premium coke requires that the electrode be heated to a temperature in the range of from about 2000°C to about 3000°C in order to provide energy to convert the carbon in the coke to a graphite crystalline form and to volatilize impurities. When a carbon body made from premium coke is heated to a temperature in the range of from about 1000°C to about 2000°C, various sulfur-containing compounds present in the coke decompose and this could result in a rapid, irreversible expansion of the carbon body. This phenomenon is termed "puffing". It is desirable to use a low sulfur containing precursor material for producing the premium coke in order to minimize or preferably eliminate problems due to "puffing".

Typically, commercially produced premium cokes are made from low sulfur containing, aromatic, slowly reacting feedstocks such as decant oils from catalytic cracking and tars obtained from the thermal cracking of decant oils and gas oils.

It would be desirable to use pyrolysis tars as feedstocks for producing premium cokes because pyrolysis tars are relatively inexpensive mixtures of aromatic compounds and have low amounts of sulfur. Generally, pyrolysis tars are heavy by-products of the cracking process for producing ethylene.

Pyrolysis tars are known to be unsuitable for the commercial production of premium coke because this production is carried out by the delayed coking process and the highly reactive pyrolysis tars convert to coke in the coils of the delayed coker furnace. This result in clogging and short operating periods.

Another drawback of the pyrolysis tars is that the premium cokes produced from them impart an undesirably large CTE to the graphite electrodes.

Prior art attempts to produce a premium coke from pyrolysis tars have the drawbacks of poor economy and/or relatively high values of the CTE.

U.S. Patent No. 3,817,853 hydrotreats a pyrolysis tar in the presence of an inert diluent and obtains a feedstock which produces graphite electrodes having a CTE of about 1.6×10^{-6} per $^{\circ}C$ and higher. While this is an improvement, a CTE of about 0.5×10^{-6} per $^{\circ}C$ or less is needed for high quality graphite electrodes. In addition, the examples in the patent teach the use of from about 12.2 to about 18.7 standard cubic meters of hydrogen per barrel of pyrolysis tar. This is a relatively high cost process.

U.S. Patent No. 4,213,846 hydrotreats pyrolysis tars, petroleum resids, and thermal tars by coking them with a hydrogen donor diluent produced by the catalytic hydrotreatment of a coker gas oil fraction generated from the delayed coking of the blend. The hydrotreated feed is an equal blend with fresh feed. This process has several drawbacks. The hydrotreated coker gas oil does not contribute to the yield of the process and the examples teach a maximum of 15% by weight pyrolysis tars.

The instant invention overcomes the drawbacks of the prior art and provides a process for the commercial production of a premium coke suitable for making high quality graphite electrodes.

In its broadest embodiment, the invention is a process for producing a premium coke for making a graphite electrode having a CTE less than about 0.5×10^{-6} per °C. comprising the steps of forming a blend of a pyrolysis tar and a hydrotreated decant oil which includes from about 50% to about 75% by weight of the pyrolysis tar and from about 50% to about 25% by weight of the hydrotreated decant oil; and coking the blend by delayed coking, whereby the premium coke is formed.

In a preferred embodiment, the hydrotreated decant oil is produced by hydrotreating a decant oil until there is added from about 2 to about 4 hydrogen atoms per average molecule of the decant oil, more preferably from about 2 to about 3 hydrogen atoms.

Another preferred embodiment of the invention is a graphite electrode made from the premium coke of the invention.

Further embodiments and advantages of the invention will be set forth in the following specification and will be obvious therefrom.

A pyrolysis tar as used herein and according to the prior art is generally the heaviest by-product of olefins production by vapor-phase cracking of liquid hydrocarbons in the presence of steam at temperatures of from about 760°C to about 930°C at pressures from about 100 pa to about 200 pa. It is the fraction which boils above about 200°C.

A decant oil as used herein and according to the prior art is generally the highest boiling by-product of gasoline production by catalytic cracking after the removal of catalyst particles by settling. It generally boils at a temperature above about 300°C.

Preferably, the pyrolysis tar used in the invention should have a sulfur content of less than about 1% by weight and the decant oil used in the invention should have a sulfur content of less than about 2% by weight. The hydrotreatment of the decant oil provides the additional incidental advantage of hydrodesulfurizing the decant oil so that the potential problem of puffing is reduced or eliminated even though the hydrotreatment is not carried out for that purpose.

Generally the hydrotreatment of the decant oil can be carried out in accordance with the prior art by contacting the decant oil with hydrogen at an elevated temperature and high pressure in the presence or a suitable catalyst.

For a fuller understanding of the nature and objects of the invention, reference should be had to the following detailed description, taken in connection with the accompanying drawings, in which:

Fig. 1 is a simplified block system of a bench-scale delayed coking unit used in a laboratory; and

Fig. 2 is a simplified block system of a pilot plant delayed coker.

Illustrative, non-limiting examples of the practice of the invention are set out below. Numerous other examples can readily be evolved in the light of the guiding principles and teachings contained herein. The examples given herein are intended to illustrate the invention and not in any sense to limit the manner in which the invention can be practiced. The parts and percentages recited therein and all through this specification, unless provided otherwise, refer to parts by weight and percentages by weight.

EXAMPLE 1

A gas oil-based pyrolysis tar A having the properties shown in Table 1 was blended with a hydrotreated decant oil A, having the properties shown in Table 2. Three blends were prepared with the amounts of pyrolysis tar A being 25%, 50%, and 75% by weight.

TABLE 1

PYROLYSIS TAR A

Specific Gravity, 60°F	1.1238
Distillation	
IBP	230°C
5%	280°C
10%	290°C
20%	320°C
30%	336°C
40%	364°C
50%	390°C
60%	450°C
70%	
80%	
90%	
95%	
EP	
% Recovery	67.5
Molecular weight	319
Carbon, wt. %	91.7
Hydrogen, wt. %	6.78
Nitrogen, wt. %	0.21
Ash, wt. %	0.0018
Metals, ppm	
Sulfur, wt. %	1.20
Modified Conradson Carbon, wt. %	23.1
Aromaticity, wt. %	
Proton NMR	40.09
^{13}C NMR	80.6
Pour Point	19°C
Flash Point	127°C
Bromine No.	15.44
Viscosity, centistokes	
38°C (100°F)	4079
99°C (210°F)	206
TGA Pitch Fraction (550°C+)	29.6 wt. %
Toluene Insoluble, wt. %	4.6
Heptane Insoluble, wt. %	21.1

TABLE 2

HYDROTREATED DECANT OIL A

Specific Gravity	1.0187
Distillation	
IBP	229°C
5%	290
10%	318
20%	346
30%	363
40%	377
50%	389
60%	404
70%	424
80%	449
90%	502
95%	-
EP	
Recovery %	90%
Molecular Weight	
Carbon, Wt. %	90.59
Hydrogen, wt. %	9.29
Nitrogen, wt. %	0.13
Sulfur, wt. %	0.37
Modified Conradson Carbon, wt. %	2.63
Aromaticity, Proton NMR	20.5
^{13}C NMR	57.6
Pour Point	-13°C
Flash Point (Open Cup)	138°C
Bromine No.	6.47
Viscosity, centistokes	
38°C (100°F)	69.42
99°C (210°F)	5.65
TGA Pitch Fraction (550°C+)	48.7
Toluene Insoluble, wt. %	0.20
Heptane Insoluble, wt. %	0.01
Hydrogen atoms per average molecule	3

A bench scale delayed coking unit as shown in Fig. 1 was used to coke each of the blends as well as separate portions of the pyrolysis tar A and the hydrotreated decant oil A.

The coking unit of Fig. 1 operates as follows:

A feed liquid 1 in tank 2 is pumped through line 3 by pump 4 at a rate of from about 17 to about 24 g. per minute. The feed liquid 1 in line 3 is conveyed to heated, pressurized coil 6 which maintains a high pressure due to pressure unit 7. The material in coil 6 communicates through line 8 to top of heated, pressurized tank 9. The temperature and pressure of coil 6 and tank 9 were about 475°C and about 689kPa. The feed period was from about 140 to about 170 minutes. After the feeding was completed, the coke was further devolatilized by heating at about 50°C per hour to about 500°C and holding this temperature for from about 75 to about 90 minutes. A pressure control valve 11 is provided for the removal of distillates and cracking gases.

For each blend, additional heating at about 1000°C was carried out and the yields for these examples is shown in Table 3. The values shown in the Table 3 are based on measurements and deviate slightly from the sum of the components, being equal to 100%.

TABLE 3

COKING YIELDS

Wt. % Pyrolysis Tar A in Blend	Feed Rate g/min	Yields Wt. % 500°C Coke	1000°C Coke	Distillate	Cracking Gas
0	24	27	25.5	64.5	8.5
25	17	31.5	29.5	60	8.5
50	21	34	32	59	7
75	17	40	37.5	53	7
100	20	39	36	56	5

The Table 3 shows that the distillates and cracking gas yields reduced as the amount of pyrolysis tar increased.

The coke from each of the tests was used to produce graphite electrodes in accordance with conventional testing procedures. The procedure used is generally as follows:

The coke which had been calcined at 1000°C was crushed and milled to 55% ± 10% through 200 mesh to obtain a flour. The flour was made into a rod about 130 mm long with a 19 mm diameter.

The rod was then converted into a graphite electrode. Typically, the last graphitizing temperature is in the range of from about 2800°C to about 3000°C.

The value of the longitudinal CTE of each rod was measured in the temperature range of from 30°C to 100°C. Only longitudinal CTE is of interest herein.

Table 4 shows the values of CTE for rods made from different blends.

TABLE 4

LONGITUDINAL CTE

Wt % Pyrolysis Tar A in Blend	CTE Graphite Electrode per °C
0	0.44×10^{-6}
25	0.47×10^{-6}
50	0.49×10^{-6}
75	0.79×10^{-6}
100	0.94×10^{-6}

It is surprising that as much as about 50% pyrolysis tar A in the blend will still provide a graphite electrode having an excellent CTE. If one were to compute the expected value for the CTE on the basis of the rule of mixtures, a much higher value greater than about 0.5×10^{-6} per °C would be calculated.

The hydrotreated decant oil modifies the pyrolysis tar to allow good continuous delayed coking and to provide excellent values of the CTE for high proportions of pyrolysis tar.

The amount of hydrotreatment given to the decant oil will have an effect on the process. If the decant oil is saturated, then it will not act as a donor. The lower limit for hydrotreating the decant oil for various blends can be determined experimentally.

The Example 1 shows that high coke yields are obtained for

relatively low amounts of hydrogen. It is also advantageous economically to hydrotreat the decant oil rather than the pyrolysis tar.

EXAMPLE 2

The tests carried out in the Example 1 were carried out with the hydrotreated decant oil A of Example 1, and a predominantly kerosene-based pyrolysis tar B, having properties as shown in Table 5.

TABLE 5

PYROLYSIS TAR B

Specific Gravity	1.0835
ASTM Distillation	
IBP	171°C
5%	238
10%	249
20%	268
30%	288
40%	321
50%	371
60%	410
70%	441
80%	-
90%	-
95%	0
% Recovery	70
Molecular Weight	398
Carbon, wt. %	90.97
Hydrogen, wt. %	7.62
Nitrogen, wt. %	0.66
Ash, wt. %	0.01
Metals, ppm	0.1
Sulfur, wt. %	0.50
Modified Conradson Carbon, wt. %	16
Aromaticity, Proton NMR	42.9
^{13}C NMR	-
Pour Point	3°C
Flash Point (Open Cup)	77°C
Bromine No.	17.98
Viscosity, centistokes	
38°C (100°F)	1154
99°C (210°F)	27
TGA Pitch Fraction (550°C+)	30.1
Toluene Insoluble, wt. %	0.1
Heptane Insoluble, wt. %	19.2

Table 6 shows the yields for the different blends and Table 7 shows the values of longitudinal CTE measured for graphite electrodes made from the blends. The measured CTE'S of the graphite electrodes made from blends having 50% and 75% pyrolysis tar were lower than one would calculate based on the mixture of the two components, and one would not expect to obtain good quality graphite electrodes based on such calculations.

TABLE 6

Wt. % Pyrolysis Tar in Blend	Feed Rate g/min	Yields Wt. % 500°C Coke	1000°C Coke	Distillate	Cracking gas
0	24	27			
25	21	27.5	26	64.5	8
50	20	27.5	26	66	6.5
75	22	29	27	65	6
100	23	29	27	63	8

TABLE 7

LONGITUDINAL CTE

Wt. % Pyrolysis Tar in Blend	CTE Graphite Electrode per °C
0	0.44×10^{-6}
25	0.55×10^{-6}
50	0.50×10^{-6}
75	0.60×10^{-6}
100	0.82×10^{-6}

EXAMPLE 3

The hydrotreated decant oil A of the Example 1 and the pyrolysis tar B of the Example 2 were blended to run tests with the pyrolysis tar content 0%, 50%, 75%, and 100%.

The pilot plant delayed coker shown in Fig. 2 was used. The operation of the pilot plant delayed coker is as follows:

Feed tank 12 supplies the blend to be coked. Pump 13 moves

the blend from the feed tank 12 through line 14 to preheaters 16 and then to the delayed coker 17. Distillates and cracking gases from the coker 17 move through line 18 to fractionator 19. Heavy products suitable for recycling are pumped from the fractionator 19 through line 21 by pump 22 to the preheater 16. Light products from the fractionator 19. move through line 23 to quencher 24 where they are cooled. The light products in the quencher 24 which are suitable for recycling are pumped by the pump 22 through line 26 to the preheater 16. The light products in the quencher 24 not suitable for recycling are removed through line 27. Gases in the fractionator 19. are removed through line 28.

Table 8 shows some of the operating parameters of the pilot plant delayed coker. A pressure of about 275 K Pa was maintained, the throughput ratio was held as close to 2.0 as possible, and the furnace temperature was in the range of from about 470°C to about 500°C. The higher temperature was used for less reactive feedstocks whereas the lower temperature was used for more reactive feedstocks.

TABLE 8

Wt.% Pyrolysis Tar in Blend	Feed Rate /min.	Throughput Ratio	Yields, Wt. %			
			Coke as made	1000°C Coke	Distillate	Cracking gas
0	83	2.1	24	22.5	72	4
50	144	1.9	21	19.5	75	4
75	106	2.0	28.5	26.5	68.5	4
100	98	3.1	38	35	58	4

The coke yields increased and the distillate yields decreased for higher proportions of the pyrolysis tar B in the blends. The yield of coke for 100% pyrolysis tar B was higher than one would anticipate from the other results because for this test the throughput was much higher than the throughput used for the other tests.

Graphite electrodes were made from the cokes calcined at 1000°C and the value of the CTE of each was measured, as in the Example 1. The measured values are shown in Table 9.

TABLE 9

LONGITUDINAL CTE

Wt.% Pyrolysis Tar in Blend	CTE Graphite Electrode per °C
0	0.21×10^{-6}
50	0.37×10^{-6}
75	0.44×10^{-6}
100	1.0×10^{-6}

EXAMPLE 4

Blends were prepared of a hydrotreated decant oil B having the properties shown in Table 10 and a naphtha-based pyrolysis tar C having the properties shown in Table 11.

The coking was carried out as described in the Example 1 and test graphite electrodes were prepared. Table 12 shows the measured values of the CTE for each of the graphite electrodes. The values of the CTE for the blends were considerably less than one would expect based on the rule of mixtures.

TABLE 10

HYDROTREATED DECANT OIL B

Specific gravity	1.04
Molecular weight	309
Carbon, wt. %	89.0
Hydrogen, wt. %	8.7
Sulfur, wt. %	0.79
Modified Conradson carbon, wt. %	1.8
Aromaticity, Proton NMR %	25
Oxygen, wt. %	0.5
Added hydrogen per average molecule	2.5

TABLE 11

PYROLYSIS TAR C

Specific gravity	1.08
Carbon, wt. %	91.0
Hydrogen, wt. %	7.5
Ash, wt. %	0.002
Sulfur, wt. %	0.1
Modified Conradson carbon, wt. %	12
Aromaticity, Proton NMR %	52.4
Toluene insoluble %	0.1
Heptane insoluble %	0.1

TABLE 12

LONGITUDINAL CTE

Wt. % Pyrolysis Tar in Blend	CTE Graphite Electrode per ^{o}C
0	0.54×10^{-6}
25	0.47×10^{-6}
50	0.49×10^{-6}
75	0.74×10^{-6}
100	1.78×10^{-6}

EXAMPLE 5

A pyrolysis tar D having the properties shown in Table 13 and hydrotreated decant oil A were blended together for coking in the pilot plant delayed coker. Blends having 0%, 50%, 75%, and 100% pyrolysis tar were used.

TABLE 13

PYROLYSIS TAR D

Specific Gravity	1.1313
Distillation	
IBP	-
5%	160°C
20%	172
30%	191
40%	207
50%	232
60%	268
70%	-
80%	-
90%	-
95%	-
EP	-
Recovery, %	55.0
Carbon, wt. %	93.1
Hydrogen, wt. %	6.8
Nitrogen, wt. %	0.0
Ash, wt. %	0.02
Metals, ppm	0.1
Sulfur, wt. %	0.22
Modified Conradson Carbon, wt. %	25.1
Aromaticity, Proton NMR	48.3
^{13}C NMR	84.3
Pour Point	14°C
Flash Point (Open Cup)	146°C
Bromine No.	12.88
Viscosity, centistokes	
38°C	14,456
99°C	80
TGA Pitch Fraction (550°C+)	40.6
Toluene Insoluble, wt. %	4.1
Heptane Insoluble, wt. %	22.8

Some of the operating parameters of the pilot plant delayed coker are shown in Table 14. The coking yields increased for larger proportions of pyrolysis tar.

TABLE 14

Wt.% Pyrolysis Tar in Blend	Feed Rate g/min.	Throughput Ratio	Yields, Wt. %			
			Coke As Made	1000°C Coke	Distillate	Cracking Gas
0	83	2.1	24	22.5	72	4
50	113	2.0	34	31	54	7
75	91	2.0	36.5	33	57	5
100	60	2.0	46	42.5	48	6

Test graphite electrodes were made and the measured values of the CTE are given in Table 15.

TABLE 15

LONGITUDINAL CTE

Wt.% Pyrolysis Tar in Blend	CTE Graphite Electrode per °C
0	0.20×10^{-6}
50	0.30×10^{-6}
75	0.47×10^{-6}
100	0.65×10^{-6}

EXAMPLE 6

Blends were made with pyrolysis tar D and decant oil C, having the properties shown in Table 15 to show the results of blends which are not in accordance with the invention.

The bench scale delayed coker of the Example 1 was used for blends of 0%, 50%, 75%, and 100% pyrolysis tar. Table 17 shows some of the operating parameters. The relatively high level of sulfur for the graphite electrode made from an equal blend would be expected to present puffing problems and would be regarded as unacceptable. This high amount of sulfur is due to the omission of the hydrotreatment which would reduce the sulfur content of the decant oil.

TABLE 16

DECANT OIL C

Specific Gravity, 60°F	1.0029
Distillation	
IBP	230°C
5%	310
10%	354
20%	372
30%	380
40%	390
50%	395
60%	410
70%	420
80%	447
90%	465
95%	493
% Recovery	95%
Molecular Weight	275
Carbon, wt. %	89.6
Hydrogen, wt. %	9.48
Nitrogen, wt. %	0.30
Ash, wt. %	0.003
Metals, ppm	N/A
Sulfur, wt. %	1.46
Modified Conradson Carbon, wt. %	2.0
Aromaticity, Proton NMR	17.4
^{13}C NMR	57.0
Pour Point	18°C
Flash Point (Open Cup)	149°C
Bromine No.	15.4
Viscosity, centistokes	
38°C (100°F)	51
99°C (210°F)	5
TGA Pitch Fraction (550°C+)	4.8
Toluene Insoluble, wt. %	0.0
Heptane Insoluble, wt. %	0.4

TABLE 17

Wt.% Pyrolysis Tar in Blend	Feed Rate g/min	Yields, Wt. % 500°C Coke	1000°C Coke	Distillate	Cracking Gas	% Sulfur in Coke
0	20	26	23.5	62	12	1.3
25	18	29	26.5	61	10	0.9
50	19	32	30	60	8	0.65
75	19	39	36	54	7	0.35
100	23	46	42.5	48	6	0.2

Graphite electrodes were made from the blends except of the blend containing 25% pyrolysis tar. Table 18 shows the measured values of CTE.

TABLE 18

GRAPHITE ELECTRODES NOT ACCORDING TO THE INVENTION

Wt.% of Pyrolysis Tar in Blend	Graphite Electrode CTE Per ^{o}C
0	0.49×10^{-6}
50	0.57×10^{-6}
75	0.81×10^{-6}
100	0.83×10^{-6}

EXAMPLE 7

The tests carried out in the Example 6 were repeated in a pilot plant delayed coker for blends containing 0%, 50%, and 100% pyrolysis tar D. In addition, the decant oil C was hydrotreated until there was added about 2.5 hydrogen atoms per average molecule of decant oil. A blend of 50% of this hydrotreated decant oil with 50% pyrolysis tar D was also coked in the pilot plant coker. Table 19 shows operating parameters and coke yields. Results for the blend containing 50% hydrotreated decant oil are shown by 50.

TABLE 19

Wt.% Pyrolysis Tar in Blend	Feed Rate g/min	Throughput Ratio	Yields, Wt. % Coke As Made	Coke 1000°C	Distillate	Cracking Gas
0	113	2.5	31	29	59	9
50	106	2.0	35	32	60	5
75	83	2.0	37.5	34	54	6
100	60	2.0	46	42.5	48	6
50*	106	1.8	27.5	26	62	7

TABLE 20

LONGITUDINAL CTE

Wt% Pyrolysis Tar in Blend	Graphite Electrode CTE Per °C
0	0.20×10^{-6}
50	0.50×10^{-6}
75	0.65×10^{-6}
100	0.75×10^{-6}
50*	0.35×10^{-6}

Table 20 shows that cokes made from the blends containing untreated decant oil have CTE's in accordance with those calculated by the rule of mixtures, whereas coke from the blend containing 50% hydrotreated decant oil has a CTE substantially lower than that calculated from the rule of mixtures.

We wish it to be understood that we do not desire to be limited to the exact details shown and described herein, or other modifications that occur to a person skilled in the arts.

Having thus described the invention, what we claim as new and desired to be secured by Letters Patent, is as follows:

C L A I M S

1. A process for producing a premium coke for making a graphite electrode having a CTE less than about 0.5×10^{-6} per ^{o}C, comprising the steps of: forming a blend of pyrolysis tar and a hydrotreated decant oil which blend includes from about 50% to 75% by weight of the pyrolysis tar and from about 50% to about 25% by weight of the hydrotreated decant oil; and coking the blend by delayed coking, whereby the premium coke is formed.

2. The process of claim 1, wherein the hydrotreated decant oil is produced by hydrotreating a decant oil until there is added from about 2 to about 4 hydrogen atoms per average molecule of the decant oil.

3. The process of claim 1, wherein the hydrotreated decant oil is produced by hydrotreating a decant oil until there is added from about 2 to about 3 hydrogen atoms per average molecule of the decant oil.

4. A graphite electrode made from the premium coke produced by claim 1.

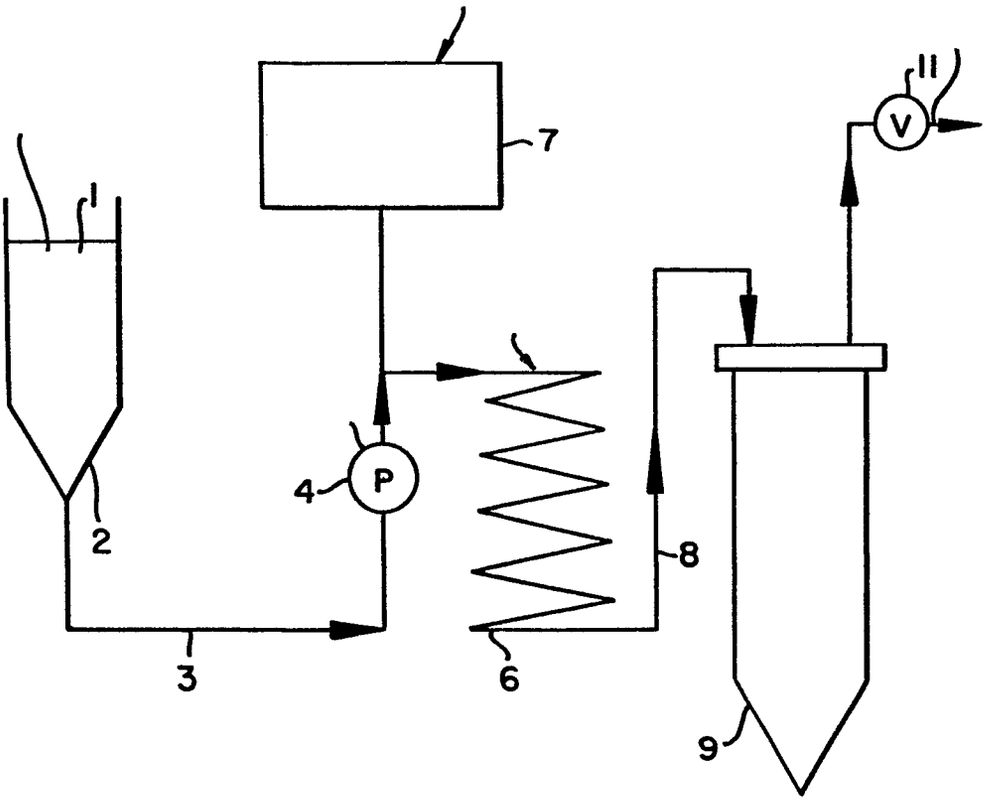

FIG. 1

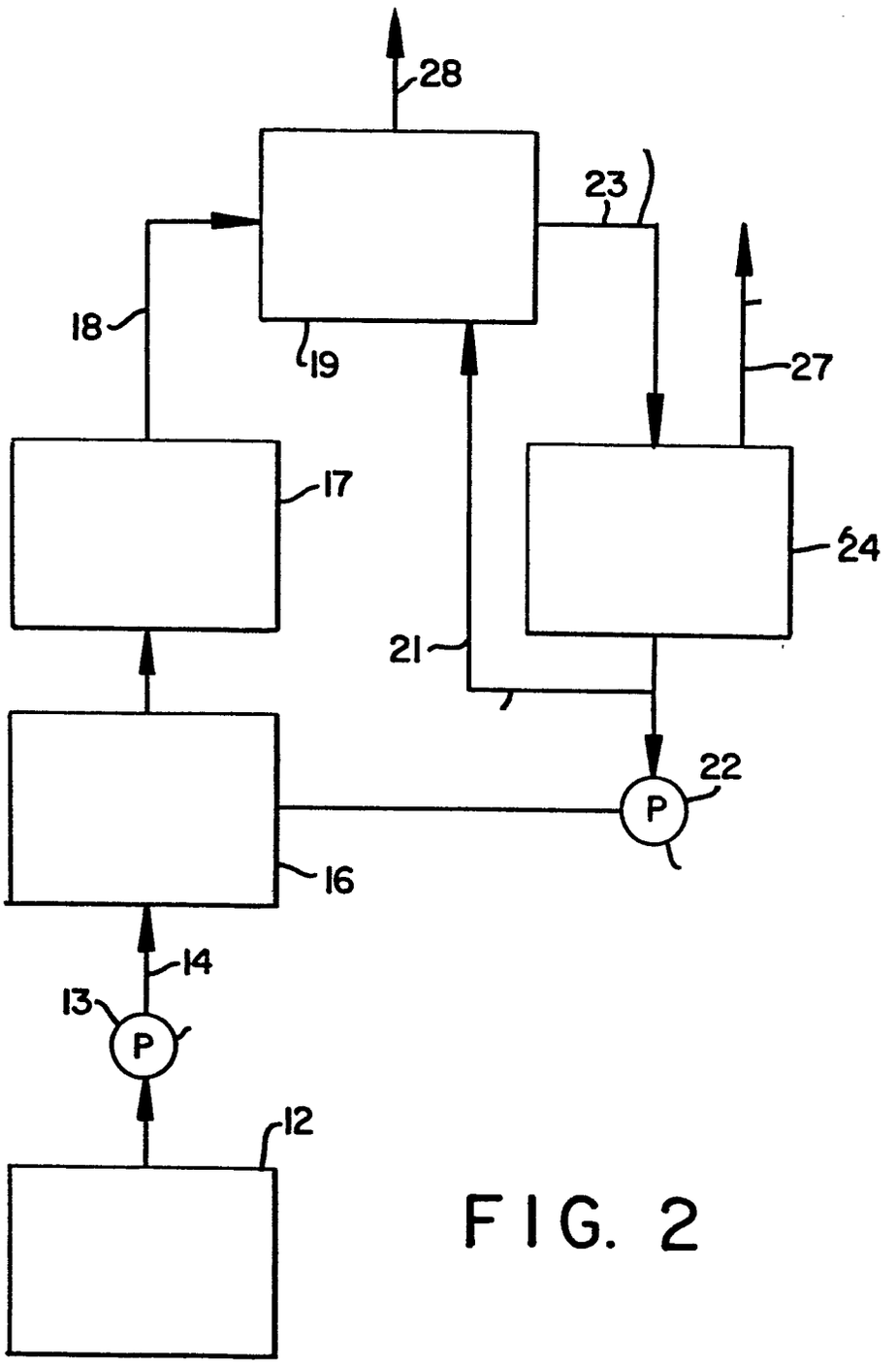

FIG. 2