High β-Resin Pitch from Coal Tar Using Submerged Combustion Flame

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Introduction

Sophisticated uses of coal tar are attractive target to producers of coal tar.

We treated coal tar using a submerged combustion technique to obtain a pitch suitable for making graphite electrodes or containing a large amount of β-resin and crack tar to increase the yield of C_{10}-C_{14} components such as naphthalene and anthracene.

Experimental

We treated the coal tar from low operation coke ovens using a submerged combustion technique which brought it into contact with a flame (1000-1300°C, for 5-30 minutes) in the reactor shown in Figure 1. Raw tar was added to the reactor during the tar treating in various conditions.

The treated tar yielded pitch by vacuum distillation. We compared the characteristics of several types of pitch made from the treated tar.

The volatile matter was trapped with coolers and a scrubber, and then the components of the volatile matter were analyzed.

Results and Discussion

Pitch for Graphite Electrodes

It was proved that the quantitative characteristics of the pitch were independent of the flame temperature, but were dependent on the final temperature of the tar in this treatment. The QI, BI, C/H and the mean molecular weight (MW) of the pitch increased as the final temperature increased (Figures 2 and 3).

Therefore, the characteristics of the pitch could be controlled by controlling the final temperature in this treatment, even if raw tar was added during the tar treating.

The pitch used for graphite electrodes (pitch IV), which was made from high operation coke ovens, has metaphase QI, while the common heat treated pitch (pitch I) has mesophase QI. Pitch I and pitch IV differ in some of their characteristics, such as C/H of QI (Table 1).

The pitch resulting from our treatment method (pitch II and III) resembled pitch IV in those characteristics. Furthermore, observations of QI particles using SEM showed that pitch II and III have metaphase QI, with 1μm diameter.

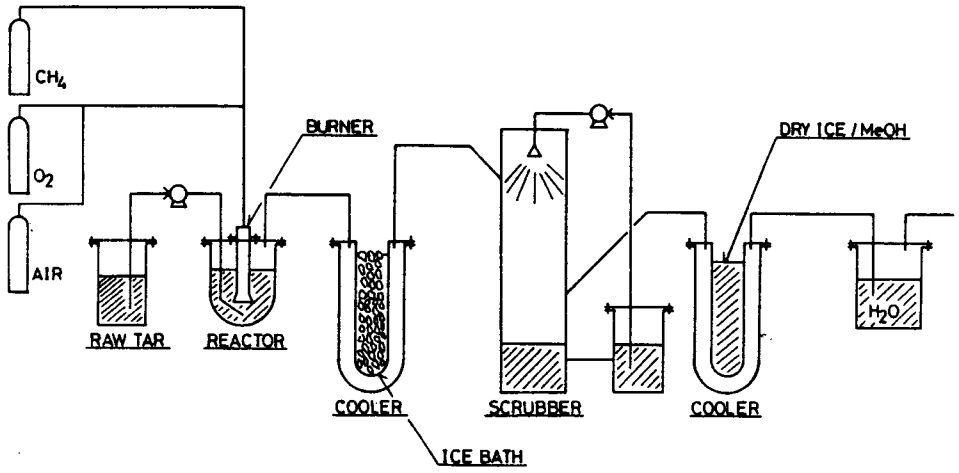

Figure 1. Tar Treatment Apparatus Using Submerged Combustion.

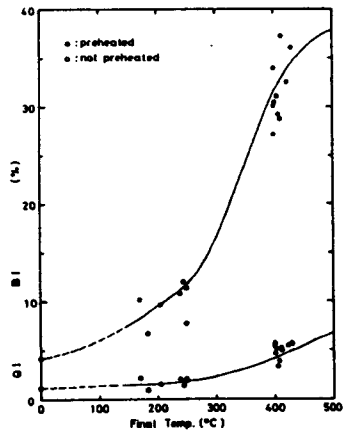

Figure 2. QI, BI Content of Pitch.

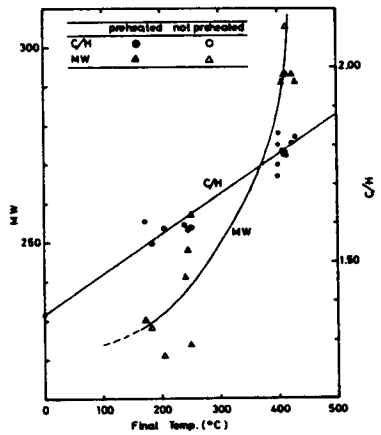

Figure 3. C/H and Mean Molecular Weight of Pitch.

Therefore, our treatment yields pitch suitable for making graphite electrodes from otherwise unsuitable tar, processed conventionally. The high temperature flame used in this treatment may cause vapor phase polycondensation of tar.

High β-Resin Pitch

Pitch with high concentration of β-resin could be obtained using our treatment in a few minutes. The rate of BI formation using our method was about 200 times as fast as the conventional method.

The β-resin content of the pitch was usually about 30% with our treatment. We tried to increase the amount of β-resin to about 60% and examined the high β-resin pitch as a pitch for carbon fiber.

Tar Cracking

The high temperature flame used in this treatment can cause tar cracking and the evaporation of the light components in the tar. We trapped more than 60% of this volatile matter, mainly with a scrubber using toluene as solvent. We compared the components of the volatile matter with the distillate of coal tar using GLC (Table 2). For example, the volatile matter had about 1.4 times as much naphthalene as the distillate, indicating that part of the tar was cracked and converted into light components by high temperature flame.

Conclusion

In our treatment, part of the coal tar is cracked to form light components and the rest polycondenses to form BI and QI.

In terms of pitch characteristics, the quality of QI resembled metaphase QI and the rate of BI formation was very fast.

In the volatile matter, the amount of components useful for making chemicals increased.

Table 2. GLC Analysis of Light Components.

Components	Content (%)	
	Volatile Matter	Distillate of Coal Tar
Indene	5.42	2.92
Naphthalene	63.25	44.24
α-Methyl-naphthalene	1.81	1.56
β-Methyl-naphthalene	4.82	4.09
2,3-Dimethyl-naphthalene	4.22	4.09
Fluorene	4.82	4.68
Phenanthrene	7.23	10.53
Anthracene	5.42	7.80
Unknown	3.01	20.09

Table 1. Characteristics of Pitch.

	Flame Temp. (°C)	Combustion Time (min.)	Final[(3)] Temp. (°C)	Softening Point (°C)	QI (%)	BI (%)	MW (GPC)	C/H of QI
Pitch I[(1)]	——	—	——	139.3	7.95	39.9	250	2.52^{5}
Pitch II	1000	10	405	129.4	3.34	31.1	291	3.58^{4}
Pitch III	1300	30	413	152.2	5.09	37.2	310	3.70^{5}
Pitch IV[(2)]	——	—	——	121.4	10.67	33.3	248	4.02^{1}

(1) made from common heat treated tar (420°C,6hrs).
(2) used for making graphite electrodes.
(3) tar temperature at the end of combustion.