EFFECT OF PITCH SOLIDS ON PENETRATION BEHAVIOR AND BINDER PERFORMANCE

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Introduction

Recently several papers have appeared on the flow of pitch (penetration) into a bed of petroleum coke particles [1-4].

This paper addresses the relationship between the various types of solids found in coal-tar pitch and the penetration characteristics of the pitch and its performance as an anode binder.

Experimental

Penetration temperature (PT) was determined using the basic techniques of Pinoir and Hyvernat [1]. A 70-mg pellet of pitch was placed on a bed of calcined petroleum coke (0.15 x 0.30 mm) and heated at 2°C/min until all the pitch penetrated into the coke. The PT is expressed as Celsius degrees above the softening point (SP) of the pitch.

Quinoline-extracted pitch solids were mounted in epoxy and the amounts of various carbon forms were determined by reflected-polarized-light microscopy.

Evaluation of the performance of three of the pitches entailed preparing 1.25-inch-diameter anodes at the optimum binder concentration (19 wt %), measuring green and baked properties, and determining carbon loss due to air burn, CO_2 reactivity, and electrolysis.

Results

Petrographic Analyses

Fourteen coal-tar pitches, none of which contained mesophase, were used in the study (9 commercial and 5 laboratory-prepared pitches). Although the SP of all the pitches is a nominal 110°C (107-115°C), they represent a wide range of penetration temperatures - 38 to >90 degrees Celsius above the SP. The petrographic analyses, along with PT, SP, and ash values, are presented in Table I. Also in the Table, various carbon forms are grouped together as follows:

Fine Solids = Fine Pyrolytic Carbon + Natural QI

Coarse Solids = Total Solids - Fine Solids

Coal-Related Solids = Cenospheres + Coke + Coal

Vapor-Related Solids = Coarse Pyrolytic Carbon + Pitch Coke

Correlation Matrices

The data in Table 1 were cross-correlated, and the correlation coefficients of PT with the carbon forms are shown in the last column of Table 1. Note that the highest correlations with PT are given with cenosphere content, coal-related solids, and coarse solids; conversely, the lowest are with fine solids, natural QI, and fine pyrolytic carbon.

These findings agree with those of R. T. Lewis [4] who found that the concentration of coarse solids (as determined by high-temperature centrifugation of pitch) correlated with PT.

Performance of Pitches

Pinoir and Hyvernat [1] considered that pitches with a PT of 35 to 40°C exhibited outstanding wetting and penetration characteristics; 45 to 55°C, normal; and >60, poor. To determine the effect of PT on pitch performance as an anode binder, anodes were made using three commercial pitches (Nos. 2, 6, and 9) having PT values of 39, 50, and >79°C, respectively, as binders (Table 1). These pitches were chosen because they cover the entire range of penetration characteristics from outstanding to poor.

Properties of anodes prepared from these three binders are shown in Table 2; averages of six determinations of each property are given. These data were analyzed using the "two-tailed" Student t-test (95% confidence level), and it was found that the properties of anodes made with pitches having PT's of 39 and >79°C (Pitch Nos. 2 and 9) are indistinguishable. Based on the t-test, the baked density and binder carbon yield of the anode made with the 50°C PT pitch are different from those of the other two anodes. However, from a practical standpoint, the properties of all three anodes are essentially the same.

Anode performance data are given in Table 3. Since the tests were run in duplicate, the t-test was not applied. However, it is quite obvious from inspection of the data that the anode made with the poorly penetrating pitch (No. 9) performed better than the pitch exhibiting normal penetration (No. 6). The increased air-burn rate and CO_2 reactivity of the No. 6 pitch are attributed to its higher Na content (Na contents of Nos. 2, 6, and 9 pitches are 21, 330, and 4 ppm, respectively).

Conclusions

1. The coarse solids in pitch, especially the coal-derived materials, correlate well with the penetration temperature.

2. A high penetration temperature does not in itself indicate that a pitch will perform poorly as a binder. Other characteristics, for example, Na content, must be taken into account.

References

1. J. Pinoir and P. Hyvernat, Light Metals, pp 497-516, 1981.

2. P. Ehrburger, et al, 17th Biennial Conference on Carbon, pp 168-169, 1985.

3. E. A. Heintz, 17th Biennial Conference on Carbon, pp 320-321, 1985.

4. R. T. Lewis, et al, 4th International Carbon Conference, pp 28-30, 1986.

Table 1. Analysis of Pitches

Pitch Designation	1	2	3	4	5	6	7	8	9	10	11	12	13	14	*
Penetration Temp., °C	38	39	48	48	49	50	51	62	>79	>80	>82	>86	>87	>90	--
Petrographic Anal., wt %															
Natural QI	8.2	10.6	10.6	13.8	10.9	13.6	11.3	11.0	9.4	8.4	13.7	8.5	11.8	9.0	-0.21
Pyrolytic C, fine	0.5	1.2	0.9	0.4	0.5	0.4	1.4	0.4	0.9	0.9	0.6	0.8	0.6	1.5	0.22
Pyrolytic C, coarse	<0.1	0.1	0.1	0.2	0.1	0.1	0.1	0.2	0.1	<0.1	0.3	0.1	0.2	0.2	0.46
Pitch Coke	<0.1	0.2	0.1	0.1	0.2	0.3	0.5	0.3	0.4	0.1	0.3	0.3	0.3	0.5	0.59
Coke Cenospheres	0.2	0.6	0.6	0.5	0.8	0.9	0.8	1.4	1.7	1.3	2.6	1.3	1.6	1.9	0.86
Coke	0	0.1	0.1	0.2	0.2	0.2	0.2	0.4	0.3	0.3	0.8	0.2	0.5	0.4	0.71
Coal	<0.1	<0.1	0.3	<0.1	0.1	<0.1	0.1	0.1	0.1	0.1	0.1	0.2	0.1	0.2	0.45
Mineral Matter	0	0.1	0.1	0.1	0.1	0	0.1	0.1	0.2	0.1	0.2	0.1	0.1	0.2	0.72
Solids, wt %															
Total	8.9	12.9	12.7	15.3	12.9	15.5	14.5	13.9	13.0	11.3	18.7	11.7	15.2	13.9	0.25
Fine	8.7	11.8	11.5	14.2	11.5	14.0	12.8	11.4	10.3	9.3	14.4	9.4	12.4	10.5	-0.18
Coarse	0.2	1.1	1.2	1.1	1.4	1.5	1.7	2.5	2.7	2.0	4.3	2.3	2.8	3.4	0.85
Vapor Related	<0.1	0.3	0.2	0.3	0.2	0.3	0.6	0.4	0.5	0.2	0.7	0.5	0.5	0.7	0.63
Coal Related	0.2	0.7	0.9	0.7	1.0	1.2	1.1	2.0	2.1	1.7	3.5	1.7	2.2	2.5	0.85
SP, °C	108	109	110	110	110	111	107	110	111	110	115	111	110	108	--
Ash, wt %	0.06	0.19	0.32	0.13	0.27	0.26	0.19	0.19	0.30	0.21	0.89	0.22	0.41	0.45	--

* Correlation coefficient of carbon form with penetration temperature.

Table 2. Properties of Prebaked Anodes

Pitch No.	2	6	9
PT, °C	39	50	>79
Baked Density, g/cc	1.48	1.49	1.48
Binder Yield, wt %	63.5	65.7	63.2
Resistivity, microohm-m	53.6	53.2	54.3
Crushing Strength, psi	5230	5340	5360

Table 3. Performance of Prebaked Anodes

Pitch No.	2	6	9
PT, °C	39	50	>79
Air-Burn Rate @ 550°C, mg/g/hr	178	204	187
CO_2 Reactivity @ 975°C, mg/g/hr	50	59	52
Electrolysis @ 975°C, % of Theoretical	118	117	117