CHARACTERIZATION OF COKES DERIVED FROM VARIOUS INDUSTRIAL PITCH BINDERS

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Introduction

Industrial pitches are used as a binder in the manufacture of various carbon artifacts such as carbon anodes, graphite electrodes and specialty carbons. Part of the manufacturing process consists of mixing a filler material (usually calcined petroleum coke) with pitch, forming the mix into a desired shape and baking to about 850-1300°C. Binders function primarily by forming pitch coke bridges between the filler petroleum coke during the baking process; the nature of these bridges affects the properties and performance of the carbon artifact [1-2].

There is a wide range of potential feedstocks for industrial pitches. We began a long range study to determine meaningful feedstock characteristics with respect to carbon and graphite products. As part of the initial phase of the study, we carbonized 27 industrial pitches with widely different properties and characterized the pitch cokes by various means. Eventually, we hope to relate pitch coke properties to product applications; for example, can we relate pitch coke reactivity to anode core reactivity?

Experimental

Pitch cokes were prepared by heating 70 grams of pitch in 100-/300-ml nested covered crucibles at a heating rate of 10°C/hr to 600°C and 25°C/hr from 600 to 1125°C; soak time at 1125°C was 13 hours. The crucibles were packed in fluid coke and the furnace was purged with nitrogen.

Pitches were analyzed by ASTM methods and metals were determined by atomic absorption spectroscopy. Air reactivity of the pitch coke was determined by heating 4 grams of 10- x 18-mesh particles in a ceramic boat for 24 hours at 525°C in a stream of air. Helium density was determined on a 10-gram sample (minus 18-mesh) using a Quantachrome Penta-Pycnometer. Reflected polarized light microscopy was used to quantify the anisotropic or needle-like structure. The property ranges of the pitches and the resultant pitch cokes are given in Table 1.

Pitch Properties	Min.	Max.	Test ASTM D-
SP, °C	102.6	165.2	3104
TI, wt%	3.3	37.8	4072
QI, wt%	0.0	21.2	2318
Beta Resin, wt%	3.2	22.8	
CCV, wt%	43.7	71.6	2416
Ash, wt%	0.0	0.28	2415
S, wt%	0.46	2.70	
Rel. Density, 25°C/25°C	1.207	1.375	71
Vis. @160°C, cps	1110	21600	5018
Dist. to 360°C, wt%	1.0	7.1	2469
Ca, ppm	<5	68	
Na, ppm	5	263	
V, ppm	<5	7	
Pitch Coke Properties			
Yield, wt%	52.1	83.6	
S, wt%	0.32	1.57	
Air Reactivity Loss, wt%	0.8	48.1	
Density, g/cc	1.963	2.068	
Needle Structure, vol%	64	100	

Table 1. Range of Properties of 27 Pitches and Derived Pitch Cokes.

Results and Discussion

The pitch coke yields (Table 1) are higher than the Conradson coking value (CCV). This is not surprising because the heating rate in the CCV test is extremely fast (30 minutes to 900°C) compared to anode baking rates; decreasing the heating rate is known to increase the pitch coke yield [3]. Three of the 27 pitch samples were also carbonized at a rate of 60°C/hr to 1000°C; the pitch yield data for these 3 samples are given in Table 2.

Sample	CCV, wt%	60°C/hr yield, wt%	10°C/hr yield, wt%
A	56.0	61.1	69.6
B	57.3	63.4	68.9
C	67.3	71.2	78.4
Temp., °C	900	1100	1125

Table 2. Effect of Heating Rate on Pitch Coke Yield

Correlations between the 17 pitch and pitch coke properties were developed; due to limited space, a matrix of only 8 selected properties is shown in Figure 1. An r value >0.64 indicates that there is 99.9% confidence that a correlation exists [4]. Note that there are both positive and negative correlations in the matrix. An example of a positive correlation is pitch coke air reactivity with pitch sodium content and a negative correlation is pitch coke anisotropy (needle structure) with pitch QI. Figure 2 shows the scatter plots for four correlations.

Future Work

We plan to evaluate a number of the pitches in 4-inch diameter anodes and determine the microstructure of the pitch coke bridges. Consequently, the correlation between pitch, pitch coke and anodes will be developed.

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References

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	SP	QI	BR	Coke Yld	Needle	Air Rx	Coke Den	Na
SP	1.00							
QI	0.36	1.00						
BR	0.46	0.36	1.00					
Coke Yld	0.65	0.83	0.62	1.00				
Needle	-0.18	-0.93	-0.41	-0.79	1.00			
Air Rx	0.20	-0.01	0.64	0.26	-0.15	1.00		
Coke Den	-0.20	-0.92	-0.42	-0.78	0.95	-0.10	1.00	
Na	0.73	0.01	0.62	0.58	0.10	0.93	0.01	1.00

Figure 1. Correlation Matrix for Pitch and Pitch Coke Properties

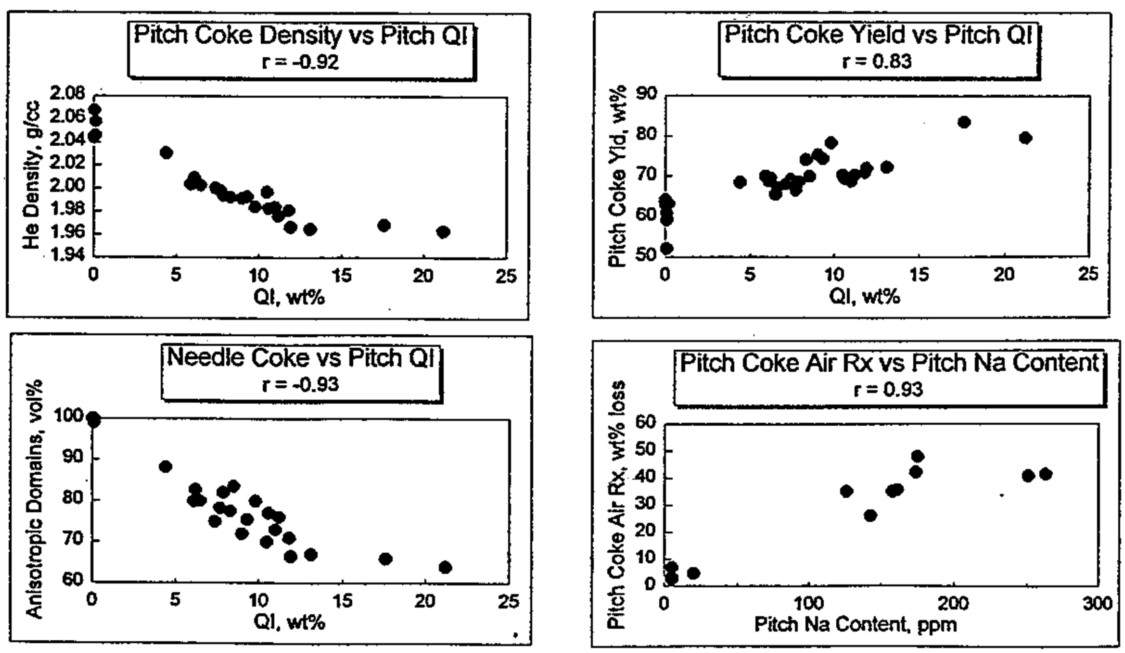

Figure 2. Correlations of Pitch Coke and Pitch Properties