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DOE/PC/91283--T11

Liquid Chromatographic Analysis of Coal Surface Properties

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Quarterly Progress Report For the Period January-March 1994

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Prepared for the United States
Department of Energy under
the Contract No. DE-FG22-91PC91283

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I. ABSTRACT

Experiments on flotation of 60 - 200 mesh treated Illinois #6 coal (PSOC-1539) and Wyodak coal (PSOC-1545) were performed during the January - March period, 1994. The coals were treated with 20-ppm alcohol aqueous solutions for 1-24 hours at the 0.002-g/min mass flow rate of alcohol aqueous solutions and at 225°C.

Flotation of Illinois #6 coal, treated with 1-propanol aqueous solution, increases with treatment durations for the first 10-hour treatment period, and then decreases with treatment durations after that period. Flotation of Illinois #6 coal, treated with isopropanol aqueous solution, increases with treatment durations for the first 18-hour treatment period, and then levels off.

Flotation of Illinois #6 coal, treated with butanol aqueous solutions, increases with treatment durations. Flotation of 1-butanol-treated Illinois #6 coal is higher than that of t-butanol-treated Illinois #6 coal. Flotation of Illinois #6 coal, treated with 20-ppm-isobutanol 20-ppm-HCl aqueous solution, increases with treatment durations for the first 10-hour treatment period, and then decreases sharply with treatment durations.

Flotation of Wyodak coal, treated with water only, increases with treatment durations. Effects of water treatment on flotation of Wyodak coal are significantly pronounced in comparison with that of Illinois #6 coal.

II. INTRODUCTION

Illinois #6 coal (PSOC-1539) was chosen as a high volatile bituminous coal, whereas Wyodak coal (PSOC-1545) was chosen as a subbituminous coal. These chosen coals were treated with 20-ppm alcohol aqueous solutions for 1-24 hours at the 0.002-g/min mass flow rate of alcohol aqueous solutions and at 225°C.

The main objectives of this research during this quarter are to identify effects of molecular structures of treatment reagents on floatability of treated coals such as Illinois #6 coal and Wyodak coal, and to examine hydrogen chloride as a catalyst for the treatment of these coals.

The coals were treated with 20-ppm aqueous solutions of various reagents such as water, 1-propanol, isopropanol, 1-butanol, 2-butanol, isobutanol and t-butanol at 0.002 g/min mass flow rate and 225°C, using nitrogen as a carrier gas for a selected solution.

Experiments on flotation of treated coals were conducted in a micro batch-type flotation apparatus at room temperature to examine change in floatability of the treated coals. Water treatment improves significantly floatability of Wyodak coal.

III. PROJECT STATUS

1. Experiments

Illinois #6 coal (high volatile bituminous coal) and Wyodak coal (sub-bituminous coal) were treated with aqueous solutions of various reagents.

Water, 1-propanol, isopropanol, 1-butanol, 2-butanol,

isobutanol and t-butanol were selected as treatment reagents. The coals were treated with 20-ppm reagents for 1-25 hours at 225°C, feeding reagent solutions into coal at 0.002 g/min. Nitrogen gas was used as a carrier gas for reagent solutions.

An empty LC column was filled with 1-g 60 - 200 mesh coal, and then placed in a GC oven to maintain the coal sample at 225°C, introducing aqueous solutions of treatment reagents in the coal-loaded column at 0.002 g/min.

Experiments on flotation of the treated coals were conducted at room temperature, using distilled water only as a flotation medium. Experiments on flotation of the coals were carried on with a 500-cc batch-type micro flotation apparatus at room temperature. Nitrogen gas was introduced at the bottom of the flotation apparatus. Distilled water was used to soak 1-g treated coal overnight prior to flotation experiments.

Experiments on adsorption of probe compounds on the treated coals were not performed during this quarter. The high pressure liquid chromatograph pump was used to feed aqueous solutions of treatment reagents into a coal-packed column during treatment experiments.

Mr. L. L. Martin, a senior chemical engineering student, worked as an undergraduate research assistant for this project during the January - March period, 1994.

2. Experimental Results

Experiments on flotation of 60 - 200 mesh treated Illinois #6 coal (PSOC-1539) and Wyodak coal (PSOC-1545) were performed

during the January - March period, 1994. These coals were treated with 20-ppm reagent aqueous solutions for 1-24 hours at the 0.002-g/min mass flow rate of reagent aqueous solutions and at 225°C.

Flotation of Illinois #6 coal, treated with 1-propanol aqueous solution, increases with treatment durations for the first 10-hour treatment duration, and then decreases with treatment durations after that period (see Figure 1). Flotation of Illinois #6 coal, treated with isopropanol aqueous solution, increases with treatment durations for the first 18-hour treatment period, and then levels off. Flotation of Illinois #6 coal, treated with water at 225°C, increases slightly with treatment durations. Flotation of Illinois #6 coal, treated with I-propanol, increases more drastically than that of isopropanol-treated one for the first 13-hour treatment duration.

Flotation of Illinois #6 coal, treated with butanol aqueous solutions, increases with treatment durations. Flotation of Illinois #6 coal, treated with 1-butanol and isobutanol, is higher than that of t-butanol-treated Illinois #6 coal (see Figure 2). These data may show that interactions between butanol isomers and functional groups such as carboxyl groups, hydroxyl groups and others on the coal surface appear to be different according to their molecular structures and reactivities at the elevated temperature.

Flotation of Illinois #6 coal, treated with 20-ppm-isobutanol 20-ppm-HCl aqueous solution, increases with treatment

durations for the first 10-hour treatment period, and then decreases sharply with treatment durations, resulting in a formation of a sharp peak on the flotation-vs-treatment duration plot (see Figure 3). Hydrogen chloride appears to act as a catalyst for the interaction between isobutanol and carboxyl group on the surface of the coal (see Figure 7).

Flotation of Wyodak coal, treated with water only, increases with treatment durations. Effects of water treatment on flotation of Wyodak coal are significantly pronounced in comparison with that of Illinois #6 coal (see Figures 1, 4 and 9). Flotation of Wyodak coal, treated with isobutanol aqueous solution, increases more drastically than that of the water-treated Wyodak coal for the first 5-hour treatment duration (see Figures 4, 5, 6 and 8). Flotation of the water-treated coal is as high as that of the isobutanol-HCl-treated coal. Hydrogen chloride does not enhance floatability of Wyodak coal, treated with 0.002-g/min-isobutanol 20-ppm 20-ppm-HCl aqueous solution at 225°C. Floatability of Wyodak coal, treated with the 20-ppm isobutanol aqueous solution at 225°C, is as high as that of untreated Pittsburgh #8 coal (see Figure 8 and Figure 12 in the July-September Quarterly Progress Report).

IV. FUTURE EXPERIMENTAL PLANS

Wyodak coal (PSOC 1545) will be treated with aqueous solutions of various reagents, which modify mildly surface properties of the coal particles at 225°C. Mechanisms on interactions between various reagents and surface functional

groups of Wyodak coal during treating the coal will be investigated with various reagents.

V. PUBLICATIONS/PRESENTATIONS

"Study on Relationships between Adsorption of Probe Compounds on Treated Coal and its Flotation in Aqueous Solutions", accepted for presentation at the AIChE 1994 Spring National Meeting, Hyatt Regency Hotel, Atlanta, Georgia, April 17-21, 1994.

"Effects of Molecular Structures of Probe Compounds on Their Adsorption on Coal", was accepted for presentation at the U.S. Department of Energy's Second Annual HBCU/Private Sector/Energy Research and Development Technology Transfer Symposium, Sheraton Civic Center Hotel, Birmingham, Alabama, April 18-22, 1994.

Table 1. Flotation of treated coals, treated with various reagents in aqueous solutions and nitrogen for 1-24 hours at 225°C.

Run #	Coal	Reagents	Concentration, ppm	Mass Rate Solution g/min	Temperature, °C	Duration, hr	Flotation, w%
T94	Illinois	ISP	20	0.002	225	24	14.73
T96	Illinois	CYCLNOL	1,000	0.002	225	26	8.16
T97	Illinois	2M1P	1,000	0.002	225	24	4.98
T98	Illinois	2M4P	1,000	0.002	225	21	6.32
T99	Illinois	2-BUT	1,000	0.002	225	24	14.09
T100	Illinois	ISO-BUT	20	0.002	225	24	15.45
T101	ADAVILLE	ISP	20	0.002	225	27	35.06
T102	Illinois	ISP	20	0.002	225	4.67	8.31
T103	Illinois	ISP	20	0.002	225	16	15.05
T104	Illinois	ISP	20	0.002	225	6	8.99
T105	Illinois	ISP	20	0.002	225	12	11.38
T106	Illinois	ISO-BUT	20	0.002	225	7	8.57
T109	Illinois	WATER	NA	0.002	225	24	8.01
T110	Illinois	WATER	NA	0.002	225	4.4	6.10
T111	Illinois	WATER	NA	0.002	225	19.5	8.54
T112	Illinois	WATER	NA	0.002	225	8.5	6.85
T113	Illinois	1-PROP	20	0.002	225	24	9.07
T114	Illinois	1-PROP	20	0.002	225	3	6.84
T115	Illinois	1-PROP	20	0.002	225	16	12.69
T116	ILLINOIS	1-PROP	20	0.002	225	5	11.91
T117	Illinois	ISP	20	0.002	225	13.25	10.25
T118	Illinois	ISO-BUT	20	0.002	225	10	10.62
T119	Illinois	ISO-BUT	20	0.002	225	14	11.90
T120	Illinois	1-PROP	20	0.002	225	10	12.68
T121	Illinois	ISO-BUT	20	0.002	225	12.5	8.51
T122	Illinois	ISO-BUT	20	0.002	225	6	10.26
T123	Illinois	ISO-BUT	20	0.002	225	4	7.16
T124	Illinois	ISO-BUT	20	0.002	225	17.5	19.50
T126	Illinois	1-BUT	20	0.002	225	25	16.97
T127	Illinois	1-BUT	20	0.002	225	16.5	13.20
T128	Illinois	1-BUT	20	0.002	225	4.5	8.34
T131	Illinois	2-BUT	20	0.002	225	3	3.75
T132	Illinois	2-BUT	20	0.002	225	8	7.70
T133	Illinois	T-BUT	20	0.002	225	24	8.72
T134	Illinois	T-BUT	20	0.002	225	14	7.93
T135	Illinois	T-BUT	20	0.002	225	3	3.75
T136	Illinois	T-BUT	20	0.002	225	6	8.12
T137	Illinois	ISP	20 +5HCl	0.002	225	14.3	5.87
T138	Illinois	ISP	20 +5HCl	0.002	175	9	5.87
T139	Illinois	ISP	20 +5HCl	0.002	150	4	0.86
T140	Illinois	ISO-BUT	20 +20HCl	0.002	225	24	26.83
T141	Wyodak	ISO-BUT	20 +20HCl	0.002	225	3	31.83
T142	Illinois	ISO-BUT	20 +20HCl	0.002	225	14	6.96
T143	Illinois	ISO-BUT	20 +20HCl	0.002	225	3	3.71
T144	Illinois	ISO-BUT	20 +20HCl	0.002	225	7	6.93
T145	Wyodak	ISO-BUT	20 +20HCl	0.002	225	13.67	5.21
T146	Wyodak	ISO-BUT	20 +20HCl	0.002	225	7	12.01
T147	Wyodak	ISO-BUT	20 +20HCl	0.002	225	24	7.21
T148	Wyodak	ISO-BUT	20 +20HCl	0.002	225	1	2.83
T149	Illinois	ISO-BUT	20 +20HCl	0.002	225	1	4.72
T150	Wyodak	ISO-BUT	20 +20HCl	0.002	225	14	6.52
T151	Illinois	ISO-BUT	20 +20HCl	0.002	225	10	15.43
T152	Wyodak	ISO-BUT	20 +20HCl	0.002	225	11.50	11.21
T153	Illinois	ISO-BUT	20 +20HCl	0.002	225	9.5	5.76
T154	Illionis	ISO-BUT	20 +20HCl	0.002	225	24	10.64

Table 1 (Continued)

Run #	Coal	Reagents	Concentration, ppm	Mass Rate Solution g/min	Temperature, °C	Duration, hr	Flotation, w%
T155	Wyodak	ISO-BUT	20	+20HCl 0.002	225	2	12.52
T156	Wyodak	ISO-BUT	20	+20HCl 0.002	225	24	10.81
T157	Wyodak	ISO-BUT	20	+20HCl 0.002	225	11.5	9.10
T158	Wyodak	ISO-BUT	20	+20HCl 0.002	225	9	14.94
T159	Wyodak	ISO-BUT	20	+20HCl 0.002	225	2	14.70
T160	Wyodak	ISO-BUT	20	+20HCl 0.002	225	1	6.56
T161	Wyodak	ISO-BUT	20	+20HCl 0.002	225	3	12.47
T162	Illinois	ISO-BUT	20	+20HCl 0.002	225	10	14.39
T163	Wyodak	ISO-BUT	20	0.002	225	13.5	3.47
T164	Wyodak	ISO-BUT	20	0.002	225	24.0	12.02
T165	Wyodak	ISO-BUT	20	0.002	225	6	5.92
T166	Wyodak	ISO-BUT	20	0.002	225	2.5	11.51
T167	Wyodak	ISO-BUT	20	0.002	225	4.5	12.94
T168	Wyodak	ISO-BUT	20	0.002	225	24	10.60
T169	Wyodak	ISO-BUT	20	0.002	225	11	2.95
T170	Illinois	ISO-BUT	20	+20HCl 0.002	225	12.5	9.98
T171	Illinois	ISO-BUT	20	+20HCl 0.002	225	11	11.54
T172	Illinois	ISO-BUT	20	+20HCl 0.002	225	8.5	13.57
T173	Illinois	ISO-BUT	20	+20HCl 0.002	225	19	9.49
T174	Wyodak	ISO-BUT	20	0.002	225	19	6.73
T175	Wyodak	ISO-BUT	20	0.002	225	9	13.90
T176	Wyodak	ISO-BUT	20	0.002	225	24.0	15.36
T177	Wyodak	water	NA	0.002	225	14.5	8.80
T178	Wyodak	ISO-BUT	20	0.002	225	6	10.39
T179	Wyodak	ISO-BUT	20	0.002	225	16.5	7.39
T180	Wyodak	water	NA	0.002	225	7	11.77
T181	Wyodak	water	NA	0.002	225	24.0	9.83
T182	Wyodak	ISO-BUT	20	0.002	225	16.0	6.74
T183	Wyodak	water	NA	0.002	225	2.0	7.05
T184	Wyodak	water	NA	0.002	225	3.0	9.72
T185	Wyodak	ISO-BUT	20	0.002	225	17.0	8.91
T186	Wyodak	water	20	0.002	225	1.0	6.21
T187	Wyodak	water	NA	0.002	225	6	14.86
T188	Wyodak	ISO-BUT	20	0.002	225	16.5	7.39
T189	Wyodak	water	NA	0.002	225	7	11.77
T190	Wyodak	ISO-BUT	20	0.002	225	1.0	4.81
T191	Wyodak	ISO-BUT	20	0.002	225	17.0	10.71
T192	Wyodak	water	NA	0.002	225	13.5	17.06
T193	Wyodak	ISO-BUT	20	+20HCL 0.002	225	19.0	10.78
T194	Wyodak	water	NA	0.002	225	21.0	15.83
T195	Wyodak	water	NA	0.002	225	24.0	24.55
T196	Wyodak	ISO-BUT	20	+20HCL 0.002	225	24.5	14.29
T197	Wyodak	ISO-BUT	20	+20HCL 0.002	225	15.0	14.21
T198	Wyodak	ISO-BUT	20	+20HCL 0.002	225	9.0	16.72
T199	Wyodak	water	NA	0.002	225	15.0	18.53
T200	Wyodak	water	NA	0.002	225	25.0	14.80
T201	Wyodak	ISO-BUT	20	0.002	225	7.5	21.62
T202	Wyodak	ISO-BUT	20	0.002	225	19.0	16.55
T203	Wyodak	ISO-BUT	20	0.002	225	24.0	20.78
T204	Wyodak	ISO-BUT	20	0.002	225	4.0	17.19
T205	Wyodak	ISO-BUT	20	0.002	225	13.0	19.53
T206	Wyodak	ISO-BUT	20	0.002	225	9.5	11.23
T207	Wyodak	ISO-BUT	20	0.002	225	20.0	24.32

ISP: isopropanol
 1-PROP: 1-propanol
 CYCLONOL: cyclohexanol

2M1P: 2-methyl 1-pentanol
 2M4P: 4-methyl 1-pentanol
 1-BUT: 1-butanol

2-BUT: 2-butanol
 ISO-BUT: isobutanol
 T-BUT: t-butanol

Figure 1. Flotation of Illinois #6 coal, treated with 0.002 g/min aqueous solution and 225°C.

Figure 2. Flotation of Illinois #6 coal, treated with 0.002 g/min 20 ppm butanol aqueous solution at 225°C, using water only as a flotation medium.

Figure 3. Flotation of Illinois #6 coal at room temperature, treated with 0.002-g/min 20-ppm-iso-butanol 20-ppm-HCl aqueous solution at 225°C.

Figure 4. Flotation of Wyodak coal at room temperature, treated with water at 225°C.

Figure 5. Flotation of Wyodak coal at room temperature, treated with 0.002-g/min 20-ppm-iso-butanol aqueous solution at 225°C.

Figure 6. Flotation of Wyodak coal at room temperature, treated with 0.002-g/min 20-ppm-iso-butanol 20-ppm-HCl aqueous solution at 225°C.

Figure 7. Flotation of Illinois #6 coal at room temperature, treated with 0.002-g/min 20-ppm-iso-butanol aqueous solution at 225°C.

Figure 8. Flotation of Wyodak coal at room temperature, treated with various 0.002 g/min aqueous solutions at 225°C.

Figure 9. Flotation of Wyodak coal and Illinois #6 coal at room temperature, treated with water at 225°C.

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