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Liquid Chromatographic Analysis of Coal Surface Properties

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Quarterly Progress Report For the Period October-December 1993

K. C. Kwon and R. R. Rigby

Chemical Engineering Department
School of Engineering and Architecture
Tuskegee University
Tuskegee, Alabama 36088

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I. ABSTRACT

Experiments on flotation of 60 - 200 mesh treated Illinois #6 coal (PSOC-1539) were performed during the October - December period, 1993. The coal was treated with water as well as 20 - 1000 ppm aqueous alcohol solutions for 3-24 hours at 150 - 225°C.

Experiments on flotation of the treated coal were conducted at room temperature, using distilled water only as a flotation medium. Flotation of Illinois #6 coal, treated with 0.002-g/min 1000-ppm methanol aqueous solution for 3 hours, increases with treatment temperatures. Flotation of Illinois #6 coal, treated with water for 3-4 hours, also increases with treatment temperatures.

Mass flow rates of 1000-ppm methanol aqueous solution, during treating Illinois #6 coal with the methanol solution for 3 hours at 225°C, increase with decreased flotation of the treated Illinois coal. However, mass flow rates of 1000-ppm n-heptanol aqueous solution, during treating Illinois #6 coal with the solution under the same treatment condition, do not affect flotation of the treated coal.

Concentrations of alcohols such as methanol and isopropanol in water do not affects flotation of Illinois #6 coal treated for 3 hours at 225°C and 0.002 g/min, whereas concentrations of isopropanol, during treating Illinois #6 coal with isopropanol aqueous solutions, increase with decreased flotation of the coal treated for 24 hours at 225°C and 0.002 g/min.

II. INTRODUCTION

The main objectives of this research during this quarter are to pursue for optimum treatment conditions of enhancing floatability of Illinois #6 coal and to examine various prospective treatment reagents by testing floatability of the treated coal in water only.

The coal was treated with water, methanol, 1-propanol and isopropanol and 1-heptanol at 150 - 225°C. Concentrations of alcohols in aqueous solutions are 5-1000 ppm. Mass flow rates of aqueous solutions during treatment experiments are 0.001 - 0.05 g/min. Experiments on flotation of treated coals were conducted in a 500-cc batch-type flotation apparatus at room temperature.

III. PROJECT STATUS

1. Experiments

Water, methanol, 1-propanol, isopropanol and 1-heptanol were selected as treatment reagents. Various treatment conditions were examined to identify optimum treatment conditions of Illinois #6 coal. Concentrations of treatment reagents in aqueous solutions vary from 5 ppm to 1000 ppm. Treatment temperatures are 150 - 225°C. Ranges of treatment durations are 3 -24 hours. Mass flow rates of aqueous solutions are 0.001 - 0.050 g/min.

An empty LC column was filled with 1-g 60 - 200 mesh Illinois #6 coal, and then placed in a GC oven to maintain the coal sample at a desired temperature, introducing aqueous solutions of treatment additives in the coal-loaded column at desired mass flow rates.

Experiments on flotation of the treated coal were carried on with a 500-cc batch-type micro flotation apparatus at room temperature, using distilled water only as a flotation medium. 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 treated Illinois #6 coal were not performed during this quarter. The high pressure liquid chromatograph pump was used to feed aqueous solutions of treatment additives into a coal-packed column during treatment experiments.

Mr. Ricardo Rigby, a senior chemical engineering student, worked as an undergraduate research assistant for this project during the October - December period, 1993.

2. Experimental Results

Experiments on flotation of 60 - 200 mesh treated Illinois #6 coal (PSOC-1539) were performed during the October - December period, 1993. The coal was treated with various treatment reagents such as water, methanol, 1-propanol and isopropanol and 1-heptanol for 3 - 24 hours at 150 - 225°C. Experimental data on flotation of treated coals were shown in Table 1.

Flotation experiments of the treated coal were conducted at room temperature, using distilled water only as a flotation medium. Flotation of Illinois #6 coal, treated with 0.002-g/min 1000-ppm methanol aqueous solution for 3 hours, increases with treatment temperatures (see Figure 1). Flotation of Illinois #6

coal, treated with 0.002-g/min water only for 3-4 hours, also increases with treatment temperatures (see Figure 2).

Mass flow rates of 1000-ppm methanol aqueous solution, during treating Illinois #6 coal with the solution, increase with decreased flotation of the coal treated at 225°C for 3 hours (see Figure 3). However, mass flow rates of 1000-ppm n-heptanol aqueous solution, during treating Illinois #6 coal with the solution, do not affect flotation of the coal treated for 3 hours at 225°C (see Figure 4).

Concentrations of alcohols such as methanol and isopropanol in water do not affect flotation of Illinois #6 coal treated for 3 hours at 225°C and 0.002 g/min (see Figure 5).

When Illinois #6 coal was treated with isopropanol aqueous solutions for 24 hours at 225°C and 0.002 g/min, concentrations of isopropanol increase with decreased flotation of the coal (see Run T89 and Run T94 in Table 1)

IV. FUTURE EXPERIMENTAL PLANS

Illinois #6 coal (PSOC-1539) and Wyodak coal (PSOC-1545) will be treated with aqueous solutions of reagents, which modify mildly surface properties of coal particles at 225°C. Mechanisms on interactions between various alcohols and surface functional groups of the chosen coals during treating the coals will be investigated with propanol isomers and butanol isomers.

V. PUBLICATIONS/PRESENTATIONS

"Investigation of Surface Properties of Coal with Probe Compounds in Aqueous Solutions", was presented at the Eighth Symposium on Separation Science and Technology for Energy Applications, Getlinburg, Tennessee, October 24-28, 1993.

The research paper, entitled "Floatability of Treated Coal in Water at Room Temperature", was submitted for publication in the Separation Science and Technology Journal.

"Adsorption of Various Probe Compounds on Coal in Aqueous Solutions", was presented at the 1993 AIChE Annual Meeting, St. Louis, Missouri, November 7 - 12, 1993.

"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 various treated coals in water only, treated with various reagents in aqueous solution and nitrogen for 3 hours at 150 - 225°C.

Run #	Coal	Reagents	Concentration, ppm	Mass Rate Solution g/min	Temperature, °C	Duration, hr	Flotation, wt%
T37	Illinois	water	NA	0.010	225	24.5	10.71
T38	Illinois	water	NA	0.010	225	25.5	8.21
T39	Wyodak	water	NA	0.010	225	25	5.74
T41	Wyodak	methanol	1,000	0.010	225	27	8.58
T46	Wyodak	methanol	20	0.010	150	23	1.38
T47	Illinois	ISP	20	0.010	225	24	16.96
T48	Wyodak	ISP	20	0.010	225	24	3.80
T53	Illinois	ISP	5	0.002	225	3	7.62
T54	Illinois	ISP	20	0.002	225	24	14.10
T59	Illinois	ISP	1,000	0.002	225	3	7.05
T64	Illinois	ISP	20	0.010	225	3	3.58
T63	Illinois	ISP	1,000	0.010	225	3	2.08
T65	Illinois	N ₂	NA	NA	225	3	3.63
T58	Illinois	methanol	1,000	0.002	225	3	5.37
T60	Illinois	methanol	1,000	0.002	225	3	7.62
T56	Illinois	methanol	100	0.002	225	3	6.45
T57	Illinois	methanol	20	0.002	225	3	4.94
T62	Illinois	methanol	20	0.010	225	3	1.77
T61	Illinois	methanol	1,000	0.010	225	3	1.84
T72	Illinois	water	NA	0.002	200	3	3.05
T65	Illinois	N ₂	NA	NA	225	3	3.63
T66	Illinois	methanol	1,000	0.005	225	3	2.58
T67	Illinois	methanol	1,000	0.001	225	3	3.88
T68	Illinois	methanol	1,000	0.010	170	3	3.04
T69	Illinois	methanol	1,000	0.002	170	3	1.70
T70	Illinois	water	NA	0.002	170	3	2.62
T71	Illinois	methanol	1,000	0.002	200	3	2.72
T73	Illinois	water	NA	0.002	150	3	2.49
T74	Illinois	methanol	1,000	0.002	150	3	1.39
T75	Illinois	heptanol	1,000	0.002	200	3	3.69
T76	Illinois	heptanol	1,000	0.002	225	3	3.58
T77	Illinois	heptanol	1,000	0.010	225	3	5.81
T81	Illinois	heptanol	1,000	0.050	225	3	4.76
T82	Illinois	heptanol	1,000	0.050	200	3	4.14
T83	Illinois	heptanol	1,000	0.050	200	3	4.81
T84	Illinois	1-PROP	1,000	0.002	225	24	4.70
T85	Illinois	heptanol	1,000	0.002	225	24	5.32
T89	Illinois	ISP	1,000	0.002	225	24	7.08
T94	Illinois	ISP	20	0.002	225	24	14.73

1-PROP; 1-propanol ISP: isopropanol

Figure 1. Effects of treatment temperatures on flotation of Illinois #6 coal, treated with 0.002-g/min 1000-ppm methanol aqueous solution for 3 hours.

Figure 2. Effects of treatment temperatures on flotation of Illinois #6 coal, treated with 0.002 g/min water for 3-4 hours.

Figure 3. Effects of mass flow rates of 1000 ppm methanol aqueous solution on flotation of Illinois #6 coal, treated for 3 hours at 225°C.

Figure 4. Effects of mass flow rates of 1000 ppm n-heptanol aqueous solution on flotation of Illinois #6 coal, treated for 3 hours at 225°C.

Figure 5. Effects of concentrations of alcohols on flotation of Illinois #6 coal, treated for 3 hours at 225°C and at 0.002 g/min in the flow rate of alcohol solutions.

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