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Liquid Chromatographic Analysis of Coal Surface Properties

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Quarterly Progress Report
For the Period January - March 1992

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I. Statement of Work

The main objectives of this proposed research work are to refine further the inverse liquid chromatography technique for the study of surface properties of raw coals, treated coals and coal minerals in water, to evaluate relatively surface properties of raw coals, treated coals and coal minerals by inverse liquid chromatography, and to evaluate flotability of various treated coals in conjunction with surface properties of coals.

Coals such as Pittsburgh seam coal, Illinois #6 coal, Wyodak coal are chosen as representatives of high-rank bituminous coal, high volatile bituminous coal and subbituminous coal, respectively. Coal minerals such as pyrite and dolomite are chosen as representative coal minerals.

Alcohols such as methanol, ethanol, isopropanol, isobutanol, tert-butanol, heptanol, 1-hexadecanol, 2-methyl-pentanol, 4-methyl-2-pentanol (methyisobutyl carbinol), n-octanol, s-octanol, and cyclohexanol as probe compounds are utilized to evaluate hydrophilicity of coals and coal minerals.

N-alkanes such as hexane, heptane and octane, and stearic acid are employed as probe compounds to evaluate hydrophobicity of coals and coal minerals. Aromatic compounds such as benzene and toluene as probe compounds are used to examine aromaticity of coal surface. Aromatic acids such as o-cresol m-cresol, p-cresol, phenol and β -naphthol are used to detect aromatic acidic sites of coal surface.

Hydrophilicity, hydrophobicity and aromaticity of surfaces for either raw coals or treated coals in water are relatively determined by evaluating both equilibrium physical/chemical adsorption and dynamic adsorption of probe compounds on various raw coals and treated coals to compare affinities of coals for water.

Dynamic adsorption experiments will be conducted in a batch reactor equipped with a magnetic stirrer. A given amount of raw/treated coals or coal

minerals, and a given amount of an aqueous solution with a known concentration of a probe compound are introduced in a batch reactor at room temperature. Concentrations of probe compounds at various contact durations are determined with a HPLC, equipped with a RI detector.

Equilibrium adsorption experiments are performed in either a batch reactor or a packed column at room temperature. A given amount of coals or coal minerals is packed in an empty HPLC column. An aqueous solution of a probe compound of interest is passed through the bed packed in the column. Concentrations of the effluent from the column are evaluated with the RI detector.

Two students are assigned for the first year to the development of precise laboratory procedures for the inverse liquid chromatography technique, using various probe compounds and coals. This involves developing procedures for the proper operation of a liquid chromatograph. Hydrophilicity, hydrophobicity and aromaticity of raw coals are detected with chromatography. Hydrophilicity and hydrophobicity of coal minerals also are determined with chromatography.

During the second year, the above students begin treating various coals and coal minerals with oxygen and nitrogen, using either a microreactor or an in-situ coal-packed column in a gas chromatograph. Hydrophilicity, hydrophobicity and aromaticity of treated coals are determined with chromatography. Hydrophilicity and hydrophobicity of treated coal minerals also are evaluated with inverse liquid chromatography.

Our research efforts should contribute significantly to the understanding of the nature of coal surface in water, and to developing further the inverse chromatography method for the evaluation of surface properties of coal in water. This work should also provide background data for improving chemical technology in coal cleaning processes.

II project Status

Research activities and efforts of the research project were idle during the period of September-December 1991, although the new research project was planned to begin in September 1991. The PI of this research project has been assigned as acting head of the Chemical Engineering Department since the Fall Semester 1991. The PI was not released from the academic duty in order to carry on this research project during the Fall Semester 1991, but the PI has been released from the academic duty (25 % FTE) in order to carry on this research project for this semester, by hiring Prof. Leo J. Hirth from Auburn University as an adjunct professor.

Several pieces of experimental equipment have been ordered, as shown in Table 1. The major equipment such as the RI detector and the solvent delivery system, placed on order in January 1992, have not been delivered. The strap chart recorder only is on hand.

The coals such as Illinois #6 coal (DECS-2; Randolph county), Adaville #7 coal (DECS-7; Wyoming state, Lincoln county), Wyodak coal (DECS-8; Wyoming state, Campbell county) and Pittsburgh #8 coal (DECS-12; Pennsylvania state, Greene county) have been obtained from the coal bank of the Pennsylvania State University (PSU). The proximate analysis and ultimate analysis of the coals were provided by the PSU coal bank, as shown in Table 2.

Most probe chemicals for the analysis of coal surface properties in water have been placed on order, as shown in Table 3. Pyrite and dolomite were selected as representative coal minerals. Graphite and activated carbon as simulating coal materials, and glass and Teflon as simulating coal mineral materials will be used for preliminary experiments and developments of experimental procedures.

III Future Experimental Plans

The PI visited the Coal Preparation Division of the Pittsburgh Energy Technology Center (PETC) to discuss future initial experimental plans of this research project with the PETC experts (Rohrer, Finseth, Gray, Jones, Rhee, Wen, Warzanski, Lai and Olson). The PETC experts suggested that initial experiments for the analysis of coal surface properties might start with graphite and activated carbon rather than raw coal. The PETC experts agreed on participation of experimental plan processes on request of the PI in order to obtain practically-useful experimental data on coal surface properties and apply these data to real coal cleaning processes. Physical characterizations of coal such as particle distributions, particle sizings, and zeta potential analysis will be accomplished with the help of the PETC experimental equipment.

Table 1 Major equipment placed on order.

<u>Equipment</u>	<u>Supplier</u>	<u>Remarks</u>
1. Cole-Parmer 250 mm strap chart with a single channel	Cole-Parmer	on hand
2. HP 1050 series isocratic pump	Hewlett-Packard	
3. HP 1047-A refractive index detector	Hewlett-Packard	
4. Rheodyne syringe-loading sample injector with 20 μ l sample loop	Rainin Instrument	
5. Eldex high pressure metering pump, 5,000 psi, 100 rpm	Rainin Instrument	
6. High pressure filter with 2 μ m filter element, 1/16" SS1 fitting connection	Rainin Instrument	
7. Adjustable back-pressure regulator, 0 -60 psi	Rainin Instrument	
8. Microsorb-MV C18 HPLC column, 5- μ m, 4.6-mm ID, 25-cm long	Rainin Instrument	
9. Empty column, 1/4"-OD, 4.6-mm ID, 250-mm long, female fitting	Alltech	
10. Rheodyne low-pressure 3-way slider valve, 1.5 mm bore, 1/4-28 threaded tube end fitting	Alltech	
11. Pressure gauge, 0-5000 psig, 1/16" tubing connection	Alltech	
12. Tee, 1/16"	Alltech	
13. Ohaus GT series precision balance, 4100 g, 0.01 g readability	PGC Scientific	

Table 2. Proximate analysis (as received), ultimate analysis (dry basis), and sulfur analysis of coals obtained from the coal bank of the Pennsylvania State University.

PSU #	DECS-2	DECS-7	DECS-8	DECS-12
Seam Name	Illinois #6	Adaville #7	Wyodak	Pittsburgh #8
Coal Rank	HVC	HVC	Subbit C	HVA
<u>Proximate Analysis</u> <u>(As received), w%</u>				
Moisture	10.43	17.34	28.42	2.40
Ash	14.47	3.45	9.90	10.00
Volatile	34.16	38.11	32.38	35.16
fixed carbon	40.93	41.11	29.30	52.44
<u>Ultimate Analysis</u> <u>Dry, w%</u>				
Ash	16.16	4.17	13.83	10.25
Carbon	65.49	73.90	64.14	74.78
Hydrogen	4.56	5.30	4.51	5.11
Nitrogen	1.11	0.99	0.86	1.23
Total Sulfur	4.52	0.98	0.73	1.12
Oxygen (Diff)	8.16	14.66	15.93	7.51
<u>Sulfur Forms, w%</u>				
Pyritic	2.27	0.10	0.28	0.38
Sulfatic	0.01	0.01	0.02	0.01
Organic	2.24	0.87	0.43	0.73
Total	4.52	0.98	0.73	1.12

Table 3 Equipment, their replacement parts and chemicals placed on order.

1/3/1992

School of Engineering and Architecture
Chemical Engineering DepartmentCole-Parmer Instrument Co.
7425 North Oak Park Avenue
Chicago, Illinois 60648
TEL: 1-800-323-4340200 SEA 4812 1000
EN 514 K. C. Kwon 8976

Line No	Cat No	Description	QTY	Unit	Unit Price	Total
1	L-08386-40	Replacement Chart Paper, 30 m long 250 mm with 0 to 100 grid, pkg/6 rolls	1	pkg	47.50	47.50
2	L-08479-30	red short replacement pens, pkg/6 pkg/25 ft	1	pkg	9.90	9.90
3	L-08373-10	Cole-Parmer 250 mm strap chart, single channel recorder	1	ea	1,190.00	1,190.00

1/10/1992

School of Engineering and Architecture
Chemical Engineering DepartmentHewlett-Packard Company
Attn: Jan Mann
P.O. Box 105005
Atlanta, GA 30348
Tel: 800-441-8360
Fax: 404-916-8285200 SEA 4812 1000
EN 514 K. C. Kwon 8976

Line No	Cat No	Description	QTY	Unit	Unit Price	Total
1	79851-A	HP 1050 series isocratic pump	1	ea	5,535.00	5,535.00
2	79851A/301	option 301, for use with HP 1050 series. modules remote control cable	1	ea	46.80	46.80
3	1047-A	HP 1047-A refractive index detector	1	ea	6,021.00	6,021.00
4	1047A/100	option 100, analog signal cable from HP 1047-A to HP 3340/92/93 integrators	1	ea	45.90	45.90
5	1047A/105	option 105, analog signal cable from HP 1047-A to universal	1	ea	45.90	45.90

3/7/1992

School of Engineering and Architecture
Chemical Engineering Department

Thomas Scientific
99 High Hill Road at I-295
P. O. Box 99
Swedesboro, NJ 08085-0099
Tel: 1-800-345-2100

200 SEA 4812 1000

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Line No	Cat No	Description	QTY	Unit	Unit Price	Total
1	2902-N70	326 Monostat aquet, reusable 1-pint squeeze bottle dispenser, 1 gallon	1	ea	18.00	18.00
2	6258-H12	822 vinyl-covered label tape, white, 1/2" wide, 500" long, pkg/6	1	pkg	18.67	18.67
3	8608-Q50	1193 stirring bar assortment, pkg of 6	1	pkg	17.00	17.00
4	9471-M61	1346 stainless steel tray, 19 1/8 x 12 5/8 x 3/4"	1	ea	25.22	25.22
5	9710-M68	1390 Qorpak glass vials with Teflon-lined closures, 60 x 17 mm, 7.4 ml, case/114	1	cs	110.15	110.15

3/11/1992

School of Engineering and Architecture
Chemical Engineering Department

Cole-Parmer Instrument Co.
7425 North Oak Park Avenue
Chicago, Illinois 60648
TEL: 1-800-323-4340

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Line No	Cat No	Description	QTY	Unit	Unit Price	Total
1	M-06256-30	wide-mouth wash bottles, 500 ml, box/6	1	bx	16.40	16.40
2	M-06406-62	1/8"-OD, 1/16"-ID, TEFLON FEP tubing, pkg/25 ft	1	pkg	11.25	11.25
3	M-06063-82	transparent polycarbonate cowboy with a spigot, 5 1/2 gallons,	1	ea	115.60	115.60
4	M-09407-40	Teflon PTFE beading, 0.078" diameter, 50' long, roll/50 ft	1	rl	38.72	38.72

3/11/1992

School of Engineering and Architecture
Chemical Engineering DepartmentRainin Instrument Company, Inc
Mack Road
Woburn, MA 01801-4628
TEL: 1-800-472-4646200 SEA 4812 5000
EN 514 K. C. Kwon 8976

Item	Cat #	Description	Quantity	unit	unit price	price
1	7125	Rheodyne syringe-loading sample injector with 20 μ l sample loop	1	ea	640.00	640.00
2	ELD-1011	Eldex high pressure metering pump, model #; B-100-S, 5,000 psi, 100 rpm, 10 lbm inlet tubing connection; 1/4-28 fittings for 1/8	1	ea	1,195.00	1,195.00
3	7160-010	right angle mounting bracket for Rheodyne sample injector	1	ea	21.00	21.00
4	R710SN	Hamilton Rheodyne injector syringe, 100 μ l	10	ea	23.00	230.00
5	219940	safety coated reagent bottle, 1000 ml, 45 mm white polypropylene screw cap with wedge-shaped inner sealing ring, case of 12	1	cs	106.00	106.00
6	AI-PCSR-2	safety-coated solvent reservoir with female 29/32 ST neck and stopper, 2 liter	3	ea	35.00	105.00
7	AI-GBSR-5	safety-coated graduated solvent reservoir with 45mm screw cap closure, 5 liter	1	ea	96.00	96.00
8	AI-SFA-PS	polyethylene 29/32 stopper with two 1/8" holes	2	ea	5.00	10.00
9	05-0108	high pressure filter with 2 μ m filter element, 1/16" SSI fitting connection.	1	ea	62.00	62.00
10	05-0107	replacement filter element, 2 μ m, pkg/2	1	pkg	25.00	25.00
11	02-0175	adjustable back-pressure regulator, a standard chemically 1/4-28 fitting connection	1	ea	98.00	98.00
12	01-0315	column fitting kits	1	ea	150.00	150.00
13	86-200-CS	Microsorb-MV C18, 5- μ m, 4.6-mm ID, 25-cm long, HPLC column	2	ea	99.00	198.00

3/11/1992

School of Engineering and Architecture
Chemical Engineering Department

ALLtech Associates, Inc.
2051 Waukegan Road
Deerfield, IL 60015-1899
TEL: 1-800-255-8324

200 SEA 4812 1000

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Line No	Cat No	Description	QTY	Unit	Unit Price	Total
1	7180	1/16" tubing cutting kit	1	ea	29.00	29.00
2	7181	spare knife file for 1/16" tubing cutting kit	1	ea	15.00	15.00
3	3206	plastic tubing cutter	1	ea	18.00	18.00
4	44701100	1 cc plastic syringe pkg/12	1	pkg	15.00	15.00
5	2429	25-mm syringe filters, PTFE membrane, polypropylene housing, 1 µm pore, pkg/100	1	pkg	90.00	90.00
6	97055	pre-cut stainless steel connecting tubing, 1/16" OD, 0.010" ID, 10 cm	6	ea	2.00	12.00
7	97065	pre-cut stainless steel connecting tubing, 1/16" OD, 0.010" ID, 30 cm	6	ea	2.75	16.50
8	97045	pre-cut stainless steel connecting tubing, 1/16" OD, 0.010" ID, 5 cm	4	ea	2.00	8.00
9	30161	1/16"-OD, 0.030"-ID, 316 stainless steel tubing	5	ft	2.00	10.00
10	28860	slip-on mobile phase filter, 2 µm, pkg/5 stainless steel	1	pkg	40.00	40.00
11	9471C	1/4"-OD, 4.6-mm ID, 250-mm long empty column, female fitting	10	ea	48.00	480.00
12	53020	Rheodyne low-pressure 3-way slider valve, 1.5 mm bore, 1/4-28 threaded tube end fitting	1	ea	60.00	60.00
13	9229	0-5000 psig pressure gauge, 1/16" tubing connection	1	ea	93.00	93.00
14	28629	1/16" tee	1	ea	30.00	30.00

3/11/1992

School of Engineering and Architecture
Chemical Engineering Department

PGC Scientific Corporation
P. O. Box 7277
Gaithersburg, Maryland 20898-7277
TEL: 1-800-424-3300

200 SEA 4812 5000
EN 514 K. C. Kwon 8976

Line No	Cat No	Description	QTY	Unit	Unit Price	Total
1	3-7146-21	Ohaus GT series precision balance, 4100 g, 0.01 g readability	1	ea	1,750.00	1,750.00
2	4-1800-13	disposable plastic weighttrays, blue, 1 5/8" x 1 5/8" x 5/16", pkg/500	1	pkg	25.95	25.95
3	29-5600	disposable aluminum dish, 43-mm diameter, pkg/100	2	pkg	7.75	15.50
4	5-2005-60	Kimax graduated low form Giffin beakers, 1000 ml, pkg/6	1	pkg	37.35	37.35
5	7-7120	black nylon beaker brush, 5" long, 2 3/8" - 2 3/8" diameter, overall 16" long	1	ea	15.36	15.36
6	19-0830	chem-dolv glassware cleaner, 1 liter	1	ea	35.00	35.00
7	19-0951-01	Kimwipes, 5" x 8 1/2", box of 280, pkg/ 60 boxes	1	pkg	99.79	99.79
8	28-4604-54	Kimble white line single graduated cylinder, SAFE-GARD bumper, 500 ml	2	ea	29.68	59.36
9	28-4604-60	Kimble white line single graduated cylinder, SAFE-GARD bumper, 1000 ml	2	ea	41.25	82.50
10	35-9480-66	Kimax filtering glass with tubulation, 2000 ml, #9 stopper	1	ea	52.48	52.48
11	36-5200-60	Pyrex fleaker, 1200 ml, pkg/10	1	pkg	103.40	103.40
12	36-9800-21	Buchner porcelain funnel with fixed perforated plate, plate diameter of 118 mm, perforated area diameter of 95 mm	1	ea	46.50	46.50
13	34-4920	Whatman filter paper, grade # 113, box/100 diameter of 11 cm	2	bx	8.05	16.10
14	71-5199-90	disposable plastic transfer pipets, 10 ml, 3-mm diameter stem, pkg/250	1	pkg	19.10	19.10
15	76-7560	stainless steel wire micro spatula, 9" long, spoon end 1 1/8" x 9/16", flat end 1 7/8", pkg/3	1	pkg	21.10	21.10
16	77-8659-11	magnetic stirring bar retriever, flexible 13" Teflon rod, white	1	ea	12.00	12.00
17	19-0984-03	Kimtex heavy duty wipers, 14" x 17", box/80, case/4 box	1	cs	86.13	86.13
18	27-0840-06	brass cork borer sets with handles, 6 pieces in set, 3/16" - 1/2"	1	set	19.20	19.20

3/27/1992

School of Engineering and Architecture
Chemical Engineering DepartmentAldrich Chemical Co., Inc.
P.O. Box 2060
Milwaukee, Wisconsin 53201
Tel: 1-800-558-9160
FAX: 1-800-962-9591200 3EA 4812 1000
EN 514 K. C. Kwon 8976

Line No	Cat No	Description	QTY	Unit	Unit Price	Total
1	27,0474-4	methanol, 99.9 + %, A.C.S. HPLC grade, 100 ml	1	ea	13.00	13.00
2	18,738-0	ethyl alcohol, 100 ml, water < 0.15 % denatured	1	ea	7.70	7.70
3	27,049-0	2-propanol, 99.5 mol % pure, HPLC grade, 100 ml	1	ea	12.00	12.00
4	32,004-8	2-methyl-1-propanol (iso-butanol), 99 +%, 25 ml ACS reagent, water < 0.1 %	1	ea	10.55	10.55
5	30,825-0	2-methyl-2-propanol (tert-butyl alcohol), 99.5 + %, HPLC grade, 100 ml	1	ea	10.00	10.00
6	H280-5	1-heptanol (heptyl alcohol), 98 %, 100 g	1	ea	7.80	7.80
7	25,874-1	1-hexadecanol, 99 %, 100 g	1	ea	7.35	7.35
8	21,401-9	2-methyl-1-pentanol, 99 %, 50 ml	1	ea	9.80	9.80
9	10,991-6	4-methyl-2-pentanol, 99 %, 100 ml	1	ea	5.40	5.40
10	29,324-5	1-octanol, 99 +%, HPLC grade, 100 ml	1	ea	17.00	17.00
11	0-450-4	2-octanol, 98 %, 100 ml	1	ea	5.40	5.40
12	10,589-9	cyclohexanol, 99 %, 1 liter	1	ea	11.30	11.30
13	13,938-6	hexane, 99 mol % pure, 500 ml	1	ea	24.20	24.20
14	27,051-2	heptane, HPLC grade, 99 %, 100 ml	1	ea	15.00	15.00
15	29,698-8	octane, 99 +%, 100 ml	1	ea	16.00	16.00
16	26,838-0	stearic acid, 99 +%, 25 g	1	ea	32.20	32.20
17	27,070-9	benzene, 99.9 +%, HPLC grade, 100 ml	1	ea	12.00	12.00
18	27,037-7	toluene, 99.8 %, HPLC grade, 1 liter	1	ea	17.00	17.00
19	24,232-2	phenol, loose crystals, 99 +%, 25 g	1	ea	10.15	10.15
20	C8,572-7	m-cresol, 99 %, 100 g	1	ea	15.75	15.75
21	C8,570-0	o-cresol, 99 +%, 100 g	1	ea	11.30	11.30
22	C8,575-1	p-cresol, 99 %, 100 g	1	ea	6.00	6.00
23	18,550-7	2-naphthol, 99 %, reagent, 100 g	1	ea	10.40	10.40
24	27,071-7	acetonitrile, 99.9 %, HPLC grade, 4 x 4 liter	1	cs	235.00	235.00

END

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9/01/92**