

LMFS-94-14
ACTIVATED CARBONACEOUS ADSORBENTS
A PRODUCTION AND TESTING STUDY

CONTRACTOR: Energy and Environmental Research Corporation
Orrville, Ohio

PRINCIPAL INVESTIGATOR: Alan Jensen, Principal Investigator
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CONTRACT AMOUNT: \$39,700

Project Schedule - 6 Months

Contract Date - 7/6/94

Start Date - 7/6/94

Completion Date - 12/1/94

Project Deliverables

Status Report - 10/1/94 ✓

Final Report - 12/1/94 ✓

OBJECTIVE / STATEMENT OF WORK

The objectives of the study are to produce and characterize test quantities of an activated carbonaceous adsorbent from North Dakota lignite. The carbonaceous adsorbent may be activated carbon or char in powdered, granular or pellet form. The carbonaceous product may be used as an adsorbent for purification of liquid or gas streams. A minimum qualification test quantity of ten pounds must be produced and supplied to the Industrial Commission. The qualification test quantities should be characterized by the appropriate chemical and physical tests to establish product specifications.

STATUS

Two tons of minus 60-mesh North Dakota lignite from The Coteau Properties Company's Freedom Mine near Beulah were collected for use in this study.

Three adsorbents were produced at the Coal Technology Corporation (CTC) two-stage 100 lb/hour pyrolysis pilot plant in Bristol, Virginia. The first adsorbent sample was carbonized under nitrogen by heating to 1150°F at a rate of 20-25°F/min and holding the charge at 1150°F for a period of 9 hours. The reactor was cooled under nitrogen. The second sample was carbonized under nitrogen by heating to 1100°F at a rate of about 18°F/min and holding the temperature at 1100°F for 6 hours. After carbonization, maintaining 5 psig of steam at 110 °F for 1-1/2 hours activated the charge. The reactor was cooled under nitrogen. The third sample was carbonized under nitrogen by heating 1020°F at a rate of about 18°F/min and holding for 2 hours at 1020°F. Maintaining 5 psig of steam at 1100°F for 4 hours activated the charge. The reactor was cooled under nitrogen. Dr. Tom Wiltowski at Southern Illinois University did characterization and analysis of the samples.

Product Characterization

<u>Property</u>	<u>Units</u>	<u>Sample # 1</u>	<u>Sample # 2</u>	<u>Sample # 3</u>	
Hardgrove index	HI	57	68	73	
BET Surface Area	m ² /g	12.34	3.54	2.21	
Total Pore Volume	10 ⁻³ cm ³ /g	18.5	17.4	15.9	
Average Pore Volume	Å	30	98	144	
SO ₂ Adsorption ¹	mg SO ₂ /g	1.18	10.23	22.81	
pH ²		10.52	9.71	9.05	
<u>Proximate Analysis</u>		<u>Raw Lignite</u>	<u>Sample # 1</u>	<u>Sample # 2</u>	<u>Sample # 3</u>
Moisture	% a.r.	34.66	1.89	4.39	5.73
Ash	% a.r.	8.50	22.07	12.52	12.87
Volatile Matter	% a.r.	29.08	12.44	23.01	26.26
Fixed Carbon	% a.r.	27.76	63.6	60.08	55.14
Heat Content	Btu/lb	6861	11367	11507	10923
<u>Ultimate Analysis</u>					
Carbon	% a.r.	41.33	64.27	65.45	63.40
Hydrogen	% a.r.	2.79	1.46	2.73	2.94
Nitrogen	% a.r.	0.77	1.15	1.15	1.11
Sulfur	% a.r.	0.60	1.14	0.59	0.56
Oxygen (by diff)	% a.r.	11.35	8.02	13.17	13.39
<u>Elemental Analysis</u>		<u>Units</u>	<u>Sample # 1</u>	<u>Sample # 2</u>	<u>Sample # 3</u>
SiO ₂	wt. % of ash		16.85	18.12	17.14
Al ₂ O ₃	"		10.21	10.45	10.43
Fe ₂ O ₃	"		14.15	14.92	14.98
TiO ₂	"		0.21	0.29	0.40
P ₂ O ₅	"		1.12	1.18	1.32
CaO	"		23.08	22.12	21.65
MgO	"		8.95	8.16	7.94
Na ₂ O	"		4.92	3.95	3.04
Na ₂ O + K ₂ O	"		5.52	4.44	3.45
K ₂ O	"		0.60	0.49	0.41
SO ₃	"		19.91	20.32	23.09

EER provided qualification samples, in excess of 10 pounds each, as a part of the project. The final report submitted by EER included preliminary process flow sheets and economic estimates for a plant using CTC's continuous (screw) reactor design to convert North Dakota lignite into activated char. Capital costs are projected to be \$11 million for a 100 tons/year facility with operating costs projected at \$38/ton.

¹ Data for SO₂ adsorption are mg of SO₂/g of adsorbent at 150E C (300E F). Measurements were done under continuous flow conditions with a simulated coal flue gas through an adsorbent bed. The flue gas was 750 ppm SO₂.

² Ten grams (10 g) of sample was mixed with 100 ml of water at 25 C for 24 hours. The mixture was filtered and the pH of the filtrate determined.