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Chemical Coal Cleaning Assessments at Gulf

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ABSTRACT

Extensive work has been conducted at Gulf Research & Development Company over the past 5 years to assess the feasibility of commercializing chemical coal cleaning technologies. Elements of this work are reviewed in this paper. It is concluded that the use of chemical cleaning will await the development of markets for premium coals and the start of new market-oriented strategies by coal companies. The trend by electric utilities toward the use of more efficient fuels and the need to pay out high cost coal reserves may force coal companies to develop premium fuels for improving market share. Significant use of chemical methods in coal preparation is a distinct possibility in the future because of developments now under way in the use of coal as an alternative hydrocarbon feedstock in chemicals and fuels applications. The potential for chemical preparation of low-ash and low-sulfur coal for these new applications is discussed.

INTRODUCTION

Interest in the development of chemical methods to prepare clean solid coal is based on the belief that new opportunities will extend the use of coal beyond its traditional boundaries of metallurgical and steam applications. If solid coal is to be substituted for oil and gas in electric power generation and for other carbon feedstocks in high-quality chemicals applications,

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there will be a market for highly refined coal whose value will be determined by factors other than heat content alone. Values anticipated for these feedstocks exceed that of steam coal (about $\$1.5/10^6$ Btu or $\$36/t$) by a large amount, thus allowing considerable latitude for technology development in the preparation of quality feedstocks from raw coal.

Recently, two interesting trends have emerged in the development of chemical coal cleaning technology. First, emphasis is now being placed more on developing deashing technology than on desulfurization. There appears to be opportunities for coal in the slurry and turbine fuels markets which require ash limits achievable by chemical methods. Since these new fuels are expected to be valued comparable to fuel oil in the $\$3.5-4.5/10^6$ Btu range, there is a realistic opportunity to develop chemical methods for preparing the clean coal components. Since the market factors appear to be more attractive and better defined for deashing than for desulfurization, it is easy to rationalize deashing research. Minerals in coal for the most part are distinct from the coal structure so that they are readily removed by chemical attack. Further, the inorganic chemistry of coal deashing is better understood than the organic chemistry of coal that has a major role in coal desulfurization. While chemical deashing is expected to be expensive, its risk is not perceived at this time to be as high as that of chemical desulfurization.

Secondly, there is a renewed emphasis on developing alternatives to hydrogen-based methods for "liquefying" the coal. This redirection appears to be caused by the realization that effective desulfurization requires extensive disruption of the coal structure. The hope is that low-sulfur and low-ash solid coal products can be prepared through intermediate solubilization without loss of useful coal characteristics. Since carbonaceous coal components of some low-rank coals exhibit a wide range of solubilities in water, caustic, and alcohols, oxidative depolymerization⁽¹⁾ and caustic solu-

bilization⁽²⁾ are being explored for their potential in preparing clean solid coal. The ideas behind these approaches are the improved reactivity of coal in the liquid phase and the possibility of precipitating useful solid coal products free from mineral matter.

Work being carried out at Gulf Research over the past 5 years to assess the feasibility of commercializing chemical coal cleaning technologies is reported in this paper. In comparing estimated costs of clean fuels prepared with chemical methods with present value of steam coals, we have concluded that the use of chemical cleaning will await the development of markets for premium coals and the start of new, market-oriented strategies by coal companies. The trend by electric utilities toward the use of more efficient fuels and the need to pay out high-cost coal reserves may force coal companies to develop premium fuels for improving market share. Significant use of chemical methods in coal preparation is a distinct possibility in the future because of developments now under way in the use of coal as an alternative hydrocarbon feedstock in chemical and fuel applications and because of impending legislation to control sulfur release to the atmosphere. The potential for chemical preparation of low-ash and low-sulfur coal for these applications is discussed.

CURRENT STATUS OF TECHNOLOGY

The primary motivation for developing chemical coal cleaning methods in the U.S. has been the need to remove organic sulfur from Eastern coals. The conventional coal cleaning methods are limited to removing inorganic sulfur forms and are based on separating coal from mineral matter containing pyritic sulfur by their specific gravity differences.

Chemical coal cleaning methods mainly rely on breaking down the bonds that connect organic sulfur groups to the coal molecules.

Because chemical processes, in comparison with conventional beneficiation methods, are expensive, their development has been slow and has not progressed beyond the laboratory or pilot-plant stage. Table I lists the prominent processes of current interest.⁽¹⁻⁷⁾ The list does not include high temperature pyrolytic/hydrogenation processes that substantially change the chemical structure and characteristics of coal.

All of the processes shown in Table I use some level of fine grinding of coal to increase surface area for exposing organic sulfur to the reagents. Acid treatment and water wash to remove 90-95% of the mineral matter is generally recommended by the process developers.

GULF WORK

Chemical coal cleaning studies at Gulf have emphasized low-cost approaches to removing sulfur and mineral matter from coal existing in the Company's Western Kentucky and Illinois reserves. Several processes have been evaluated as part of our research activities, representing considerable diversity in approach to sulfur removal. This paper discusses three approaches to dry chemical desulfurization:

1. Air/Steam Cleaning of Coal--effective at removal of pyritic sulfur.
2. The Nitrogen Dioxide Process--effective in removing organic sulfur.
3. Molten Alkali and Acid Treatment--effective in removing both sulfur and ash.

1. Air/Steam Cleaning of Coal

The air/steam process is a dry method for converting sulfur to gaseous forms.⁽⁸⁻¹⁰⁾ The process is believed to be one of the

Table I
 PROMINENT CHEMICAL COAL CLEANING PROCESSES

Developer	Process/Description	Temperature °C	Pressure Atm	Expected Removals, %			Btu Recovery %
				Total	Sulfur Organic	Ash	
Pittsburgh Energy Technology Center, DOE(1,3) Ames Laboratory (1,3)	Oxydesulfurization: Coal- water slurry is treated with compressed air.	150-200	34-102	80-90	20-50	--	80-90
	Molten alkali: Sodium car- bonate treatment of preoxi- dized coal followed by acid/water wash	350-400	3-20	88	30-38	>90	--
TRW, Inc.(4)	Molten alkali: Crushed coal is treated with molten alkali (NaOH/CaOH) and acid/water washed.	380-400	1	90-100	40-90	>95	90
Battelle(2,3)	Hydrothermal: Mineral and organic sulfur leached by sodium and calcium hydroxide solutions.	98-170	15-170	60-85	24-70	--	75-90
	Water-refined coal: Solubilizing coal in sodium hydroxide solution, followed by filtration, and recipitation of coal by acid.	250-275	13-40	60-70	20-60	>95	--
General Electric(3)	Microwave: coal suspended in sodium hydroxide solution is irradiated with microwaves	--	1	70-99	30-60	--	96
Guth (KWB); Gulf(3,15)	NO ₂ Process: Dry crushed coal is treated with NO ₂ , and caustic leached.	100	1	60-90	30-70	--	91
Jet Propulsion(5) Laboratory	Chlorinolysis and hydrodesulfurization: Chlorine is reacted with fine coal sus- pended in water. Reacted coal is treated with hydrogen to increase sulfur removal.	70; 600-700	1	80-90	40-70	--	80-85
Hazen/Wedlog(3,15)	Magnex: Magnetic separation of pyrite reacted with iron carbonyl.	150	1	80	--	--	--
Gulf(7)	Air/Steam Magnetic Separation: Dry coal desulfurized and deashed.	300-350	1	60	--	20	80-90

simplest approaches to coal desulfurization, and was optimized at the bench scale. With use of magnetic methods following the air/steam reaction, 97% of pyritic sulfur has been removed from West Kentucky No. 6 coal containing pyrites of 10-20 micron mean particle diameters. There is no firm evidence of organic sulfur removal.

Experimental

A schematic diagram of the reactor is shown in Figure 1. The unit consists of a 2" thin-walled fixed-bed reactor, surrounded by a 1/8" x 4' coil. The treating gases are passed through the coil before entering the reactor. The reactor containing coal could be immersed in and lifted out of a fluid-bed heating bath at will. The reactor effluent passes through an intermediate trap where light hydrocarbon is condensed. The gases then pass through a scrubber which contains 30% hydrogen peroxide solution to convert sulfur dioxide to sulfuric acid. This solution is analyzed for sulfate. Uncondensed gases are analyzed for COS, H₂S, SO₂, and CS₂.

In addition to measurements of sulfur evolved as gases, the coal sulfur forms were determined by ASTM D2492 method. The Leco method (ASTM D1552) was used for quick determination of total sulfur. Values were compared to those obtained by the Eschka method (ASTM D3177). Agreement between the methods for measuring total sulfur was within 2%. Sulfur balance ranged from 87 to 90%, and material balance between 97 and 98% on a moisture-free basis. The ASTM measurement of pyritic sulfur taken alone can be in error when iron oxides are present in significant quantity as is the case for these experiments.

Magnetic separation experiments were carried out on dry coal samples employing a Frantz Isodynamic Separator producing magnetic fields up to 20 kilogauss.

Results

Results are shown in Table II and are to be described in more detail elsewhere.⁽⁷⁾ The most important process variables affecting

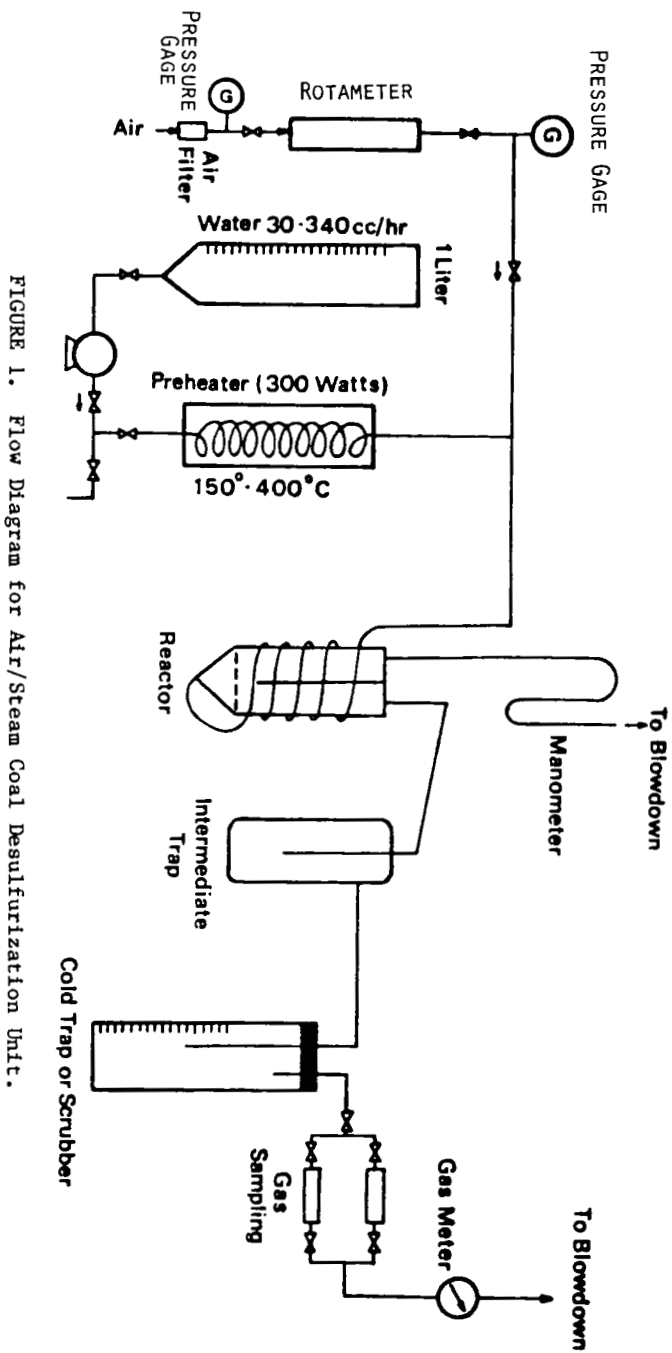


FIGURE 1. Flow Diagram for Air/Steam Coal Desulfurization Unit.

Table II

RESULTS OF AIR/STEAM CLEANING AT OPTIMIZED CONDITIONS

Temp. °C	Res. Time min	Reaction Conditions				Total Sulfur in Product Wt%	Coal Recovery Wt%	Coal Desulfurization#	
		Steam/Air g/g	Air/Rate P/h	H ₂ O Coal g/g	% of Tot. Sulf.			% of Pyritic Sulf.	
350	15	9.7	4.8	0.6	2.06 1.86*	96 88*	30 42*	47 69*	
350	45	9.7	4.3	1.8 †	2.13 1.52*	94 84*	29 55*	46 92*	
350	15	9.7	4.8	0.3	2.13	96	27	43	
350	15	4.8	9.6	0.6	1.94 1.74**	96 85**	34 48**	55 79**	
350	15	4.8	9.6	0.15	2.04	96	30	49	
300	45	4.8	9.6	0.6	2.07	99	27	43	
400	5	9.7	4.8	0.2	2.09 1.20* 1.83***	92 31* 55*	32 NA NA	51 NA NA	

* Magnetic separation at 20 kilogauss.

** Magnetic separation at 14 kilogauss.

*** Second-stage magnetic separation at 14 kilogauss.

Based on initial total sulfur content of 2.71 wt% and pyritic sulfur content of 1.55 wt%.

† Can be reduced to 0.6 or lower.

coal desulfurization and coal recovery were reaction temperature, residence time, and the steam-to-air ratio. The steam-to-coal ratio, air feed rate, and particle size of feed coal up to 8 mesh were not found to be significant.

Maximum desulfurization and recovery of coal took place within the 300 to 350°C range. There is an optimum steam-to-air ratio for maximum desulfurization (35% total sulfur removed) of coal. Desulfurization was enhanced by increasing oxygen partial pressures to a steam-to-air ratio of 9.7 by weight for reaction at 350°C (Fig. 2).

Our work has indicated that steam plays a unique role as catalyst in promoting dry oxidative desulfurization of coal. Steam appears to penetrate the coal and promote access of the oxygen in the air to the pyrites for enhancing the reaction. We did not observe desulfurization by air with inert diluting gases such as N₂ to the same significant extent as with steam. Further, moderate air/steam treatment followed by magnetic separation appears to be an attractive way to remove pyrites from coal.

Mild air/steam treatment leads to conversion of pyritic sulfur to a highly magnetic form so that this method appears to be a simple and inexpensive way to enhance magnetic desulfurization and deashing without using expensive and toxic reagents.⁽¹¹⁾ Air/steam treatment was found to convert some of the pyrite present to oxidized forms such as sulfate, pyrrhotites, and possibly magnetite. Additional sulfur reductions equivalent to 0.3-1.1 lb SO₂ per million Btu were realized with use of magnetic separation following air-steam treatment. Total sulfur was reduced from 2.71% to 1.94 by steam/air treatment alone. Steam/air and dry magnetic separation achieved a further reduction to 1.52% at 84% Btu recovery in a single-stage operation. Total sulfur reductions up to 56% have been achieved for this coal in combined air/steam and magnetic separation treatments at Btu recoveries of 84-96% when middling products are included (1.8% S, 53% yield).

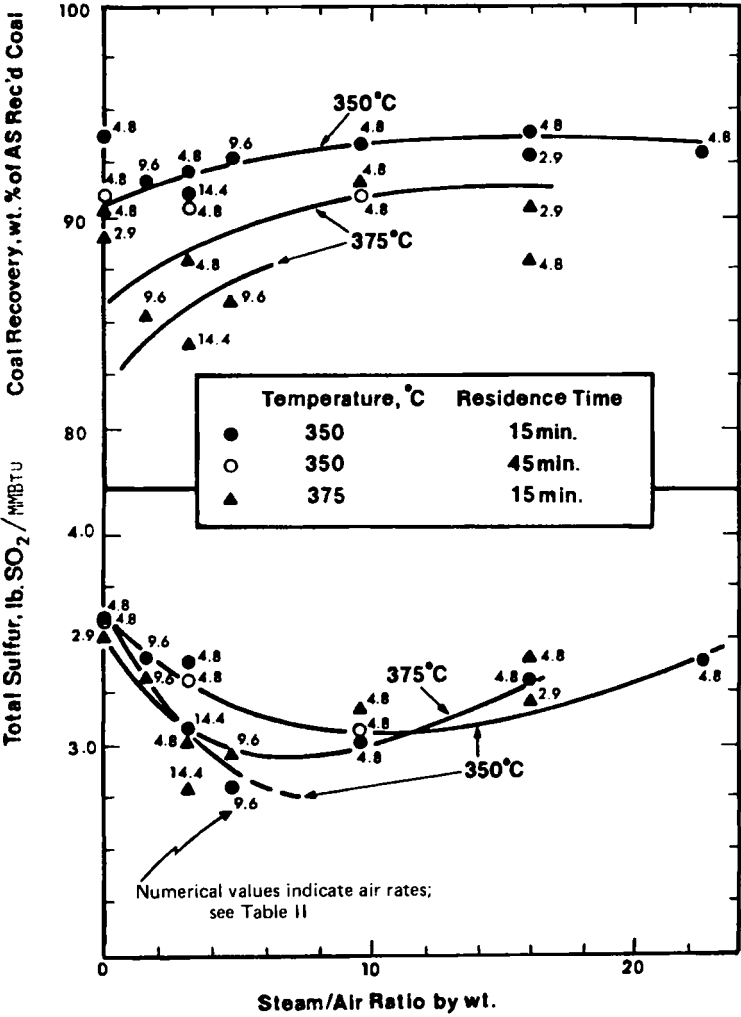
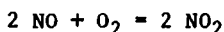
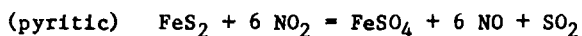
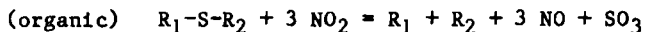


FIGURE 2.
Effects of Operating Conditions on Coal Desulfurization and Coal Recovery.

2. The Nitrogen Dioxide Process

The use of nitrogen dioxide for coal desulfurization was first reported by KVB Inc.^(12,13) Desulfurization by the process is believed to involve the following reactions:



The oxidant is recovered from the product gases and oxidized with air to effect its regeneration. Nongaseous oxidized-sulfur forms are removed from coal by water or caustic washings. The process has several variations which include the use of a carrier for the oxidant. Kentucky No. 6 constituted the principal coal used in this study.

Experimental

The coal desulfurization reactor was similar to that used for the air/steam process shown in Figure 1. The oxidizing feed gas was a dry mixture containing up to 2% nitrogen dioxide, the remainder being nitrogen. Reactor effluent passed through a trap to the nitrogen analyzer for analysis of nitrous oxide and nitrogen dioxide. A port was provided for gas sampling for gas chromatography. Temperature measurements were made in the coal bed and at other strategic points within the bath.

Reaction times varied between 1 to 4 h, depending on the gas feed rate and coal charged. Sulfur forms were determined by ASTM D 2492, total sulfur by the Eschka method, and nitrogen, carbon, hydrogen, and heating value analysis by standard procedures. Desulfurization determinations, reported here are the result of considerable work on independent systems and model compounds (including NO_2 removal of organic sulfur from coal precleaned by a microbial process which left depyritized coal free from interfering iron).

Caustic leaches were carried out at 100°C using a 1N solution of sodium hydroxide for 1 h. Typically, 10 g of oxidized coal was refluxed with the caustic solution followed by filtration, aqueous washing of the filtrate, and drying of the coal.

Results

Sulfur and nitrogen concentrations of Kentucky No. 6 coal oxidized at temperatures ranging from 50 to 350°C with and without follow-on caustic leaching are shown in Figure 3. Effective desulfurization requires 300–350°C. Oxidation of coal at temperatures in excess of 350°C incurs excessive loss of carbon and coal volatiles.

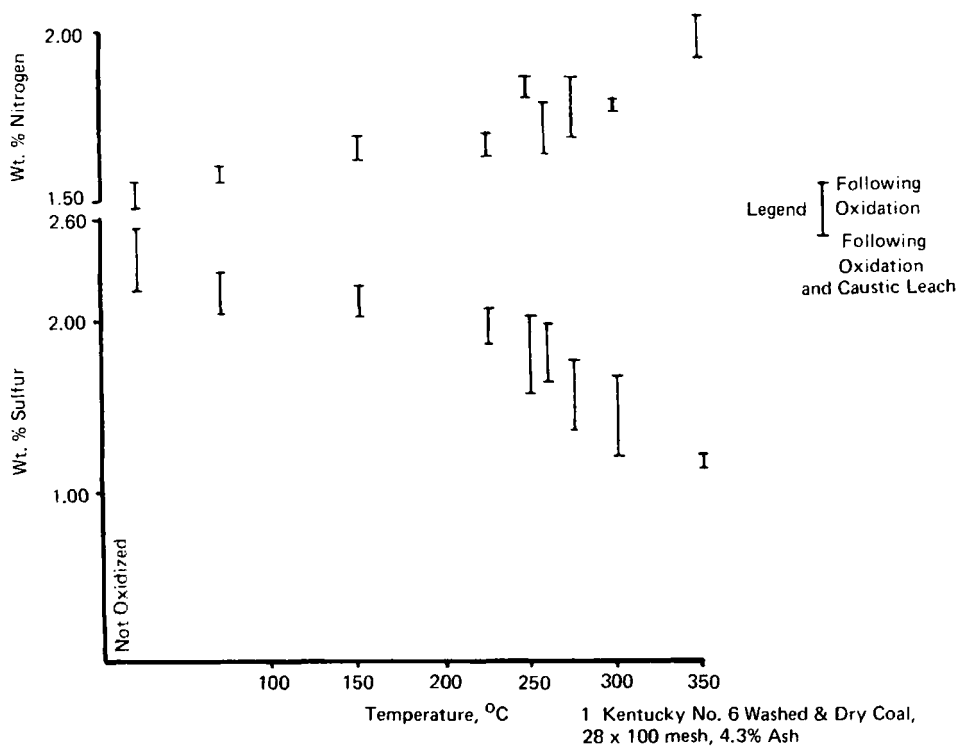


FIGURE 3. NO₂ Oxidation and Caustic Leach of Coal.

These results indicate an affinity of selected maceral and mineral species for caustic. Our results suggest that penetration of the vitrinite and exinite maceral components is followed by an explosive increase in the internal surface area for this coal which is believed to be caused by sudden loss of water and volatiles at the high reaction temperatures during molten alkali treatment. This effect may also be observed with use of microwave heating in the presence of caustic but probably would not be observed when processing caustic in water because of the high vapor pressure of the process water.

Apparently, the macerals exhibiting plastic properties play a further important role in the desulfurization process. In the plastic state at high temperatures, the solid macerals dissolve and diffusion barriers characteristic of the solid state are removed, thus promoting mixing of both organic and mineral components. The overall result of this effect is improved desulfurization and more effective demineralization of the reacted coal during acid wash.

By mechanically removing nonreactive fusinites and other inert components from the process feed, it may be possible to both improve the process economics and to produce a cleaned coal that is even lower in sulfur and ash than that now prepared by TRW.

Economics

Several investigators have estimated the costs of chemical coal cleaning processes.^(2,14,15) Gulf has analyzed and updated these estimates to 1982 costs for a typical coal cleaning plant processing 2.5×10^6 t/yr of run-of-mine mid-Western bituminous coal. The process layouts considered included a front-end physical beneficiation plant operating 13.6 h/day and 226 days/yr. Provisions were made in the analysis for stockpiling physically cleaned coal which could be withdrawn for chemical processing 24 h per day, 365 days/yr, at 90% capacity factor.

Table III

SULFUR AND NITROGEN IN NO₂ TREATED COAL*

Oxidation Temperature, °C	Not								
	Oxidized	73	150	225	250	260	300	350	
Total Sulfur, Wt%	2.54	2.29	2.20	2.06	2.02	1.98	1.77	1.19	
Total Nitrogen, Wt%	1.56	1.60	1.67	1.79	1.87	1.79	1.82	2.00	
Forms of Sulfur, Wt%									
Pyritic	1.25	1.02	1.01	0.99	0.95	0.94	0.90	0.87	
Elemental	0.01	0.01	0.02	0.01	0.0	0.0	0.0	0.0	
Sulfate	0.08	0.06	0.07	0.13	0.17	0.17	0.27	0.02	
Organic	1.20	1.20	1.10	0.96	0.90	0.87	0.60	0.30	

* Kentucky No. 6 (28 x 100 mesh) washed and dried coal was oxidized using 0.5-1.0 vol% nitrogen dioxide in nitrogen. Oxidized coal was leached with a 1N aqueous caustic solution for 1 h. Results for aqueous caustic leach for oxidized and comparative unoxidized coal are shown in Figure 3.

Table IV

EFFECT OF OXIDATION ON HEATING VALUES OF COAL*

Oxidation Temperature, °C	Heating Value,** Btu/lb	Recovery of Oxidized Coal***, %	Heating Value x (% Recovery of Coal) %	Btu Loss****
Not Oxidized	14 058	100	14 058	0
200	13 858	98	13 581	3.4
225	13 451	97	13 047	7.2
260	13 374	94	12 572	10.6
300	12 945	91	11 780	16.2
350	12 770	88	11 238	20.1

* Kentucky No. 6 washed and dried coal.

** After oxidation.

*** Weight % based on charged coal.

**** Based on unoxidized coal.

The feed coal used for most of the study was 28 x 0 mesh. Oxidation of finer coal to 200 mesh did not enhance desulfurization. Desulfurization achieved with NO₂ was not significantly greater than that obtained with use of the air/steam method.

3. Molten Alkali Treatment

The molten alkali process with acid treatment being investigated by TRW is claimed to have one of the highest sulfur and ash removal efficiencies.⁽⁴⁾ Two samples of Illinois coals that were physically cleaned by Gulf were sent for molten alkali/acid wash treatment by TRW. The results of the TRW test work are shown in Table V. These results indicate that total sulfur in the coal

Table V

REMOVAL OF SULFUR AND ASH FROM COAL BY TRW (GRAVIMELT) PROCESS

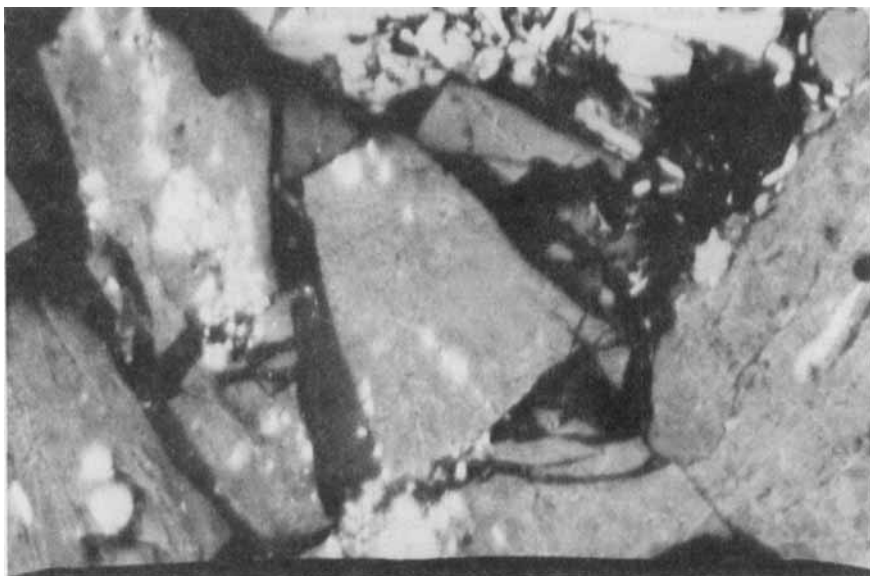
Illinois Coal	Sample ID	Weight %					Heat Content Btu/lb		lb S/10 ⁶ Btu
		Ash	S _t	S _p	S _s	S _o	Dry	MAF	
#5 Seam	Gulf	14.42	3.06	1.35	0.01	1.70	11946	13687	2.56
	29084								
	Control								
	Gulf	0.75	0.39	0.08	0.01	0.30	13806	13910	0.28
	29084	0.48	0.51	0.05	0.04	0.42	13615	13678	0.37
	Extracted								
#6 Seam	Gulf	6.83	2.86	1.05	0.01	1.80	13215	14118	2.16
	29085								
	Control								
	Gulf	0.37	0.59	0.05	0.01	0.53	13807	13859	0.43
	29085	0.39	0.77	0.06	0.01	0.70	14058	14113	0.55
	Extracted								

S_t - Total SulfurS_p = Pyritic SulfurS_s = Sulfate SulfurS_o = Organic Sulfur

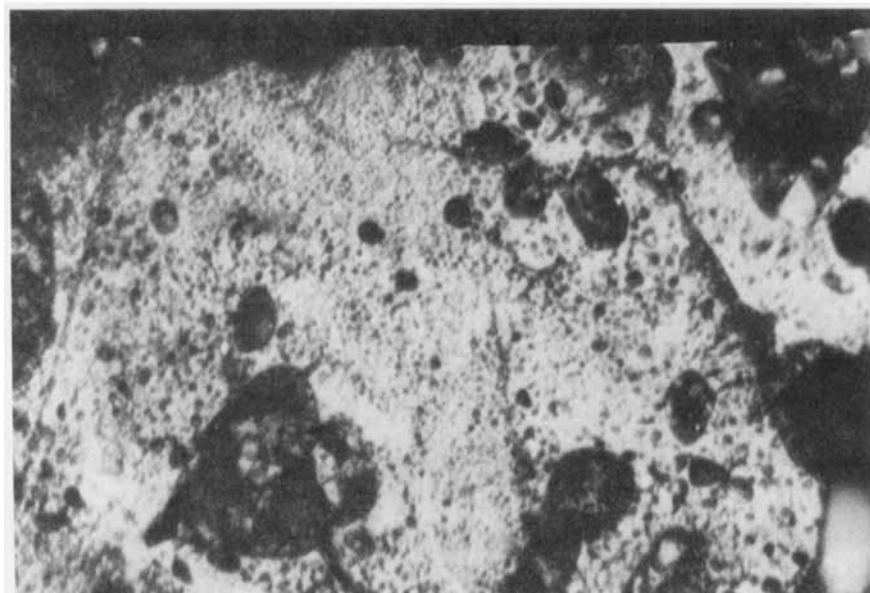
MAF = Moisture- and Ash-Free Coal

samples treated can be reduced by the molten alkali process to <0.70% and the ash content to <0.75%.

Petrographic analyses of the treated samples conducted by Gulf (Figure 4) illustrate the effect of molten alkali treatment upon the maceral and mineral components. The product material from the TRW treatment shows considerable physical distortion of the vitrinite and exinite macerals and almost complete removal of the sulfide minerals. Pseudo-vitrinite and other relatively nonreactive macerals show minimal evidence of reaction with the caustic. The fusinite or semifusinite macerals remain unreacted and inert to the molten alkali treatment.



a. Before Treatment



b. After Treatment

FIGURE 4. Molten Alkali Treatment. Petrographic Analyses of Illinois Coal Samples.

The results of this analysis indicated that the 1982 investment costs of all the chemical cleaning processes, including front-end beneficiation, range from \$160 million to \$260 million. The "dry" processes (TRW, steam/air, and KVB/Gulf) represented low investments, ranging from \$160 to \$185 million. The higher investments for the "wet" processes (e.g., PETC, JPL-2, and GE), in the range of \$180 to \$260 million, resulted from larger plant sizes and relatively more complex equipment required to handle the large quantities of water in the coal slurries.

The cost of coal lost during the cleaning process was considerable for all processes, ranging from \$24 to \$36 million per year. The overall cost of cleaning, estimated for a 20% discounted cash flow rate of return on 100% equity, ranged from \$1.5 to \$3/per million Btu of product.

Costs and performance trends for beneficiation of high-sulfur Illinois coal are compared at the conceptual level for several technologies ranging from conventional to chemical in Figure 5. The cost figures of \$2.5 to 4.5 per million Btu include raw coal cost at \$1.30/per million Btu and approximately represent the addition of acid extraction and water wash steps to the chemical cleaning processes to reduce ash levels to <1.5% in the product. The product fuel conceptualized for this study would be used in industrial or utility boilers, but no costs were included for slurry preparation.

The ash removal data shown in Figure 5 were incidental to the primary goal, which was desulfurization. Processes developed specifically for deashing may exhibit a cost performance trend curve different from that of Figure 5. In comparison, coal liquids can be taken as a reference case for chemical coal cleaning technologies. Our evaluation indicates a considerable potential for chemical pathways other than high temperature hydrogenation to prepare suitable clean coal at costs considerably below that anticipated for coal liquids at >\$6/million Btu.⁽¹⁶⁾

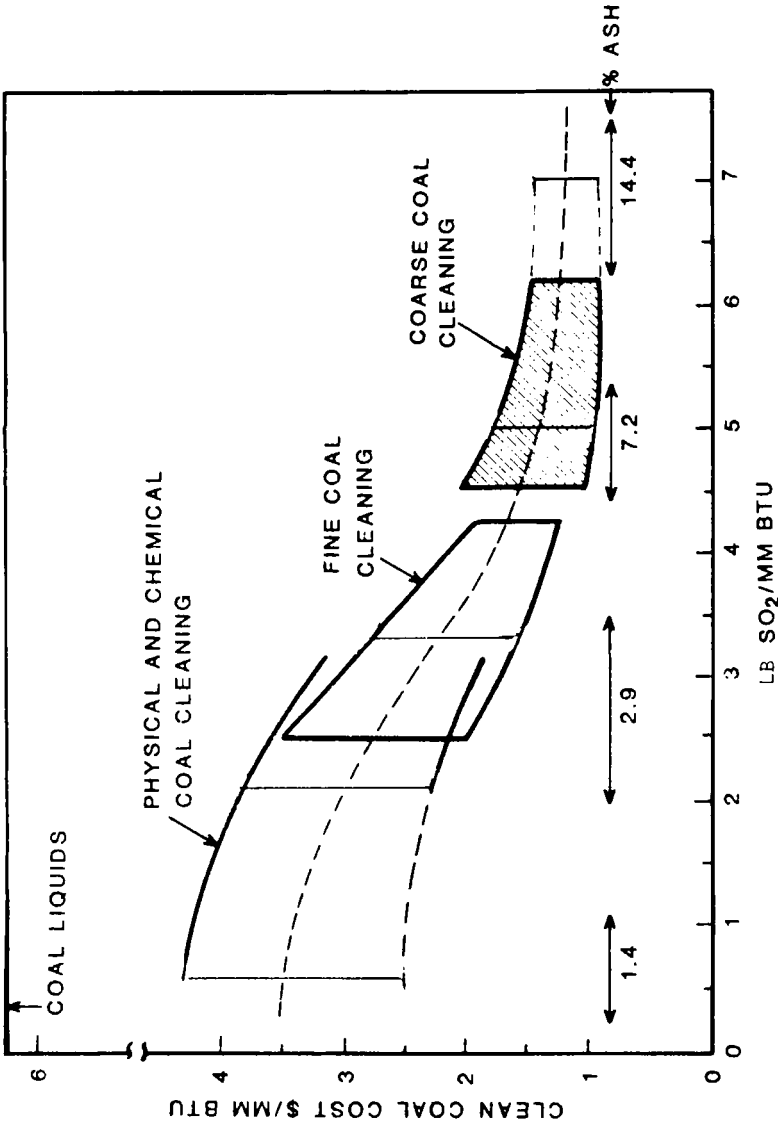


FIGURE 5. Cost of Coal Cleaning Technology in 1982.

CONCLUSIONS

- Chemical methods, as now developed at the laboratory level, can prepare coals of quality adequate for coal slurry and turbine applications.
- Chemical methods will require development of process technology more sophisticated than now used in coal preparation.
- Solid clean fuels prepared by chemical methods may cost \$2.5-4.5/million Btu. Coal liquids preparation will likely involve additional costs anticipated at >\$2.5/million Btu.
- There is an interesting rationale evolving towards development of nonhydrogen-based coal liquefaction technologies directed at solubilizing coal components for enhanced desulfurization in the liquid state and for more effective deashing by filtration. Water, caustic, and alcohol systems are being studied.

ACKNOWLEDGMENT

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