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TECHNICAL REPORT

September 1, 1995, through November 30, 1995

Project Title: **DESULFURIZATION OF COAL: ENHANCED SELECTIVITY USING PHASE TRANSFER CATALYSTS**

DOE Cooperative Agreement Number: DE-FC22-92PC92521 (Year 4)
ICCI Project Number: 95-1/1.1D-3P
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ABSTRACT

Due to environmental problems related to the combustion of high sulfur Illinois coal, there continues to be interest in the development of viable pre-combustion desulfurization processes. Recent studies by the authors have obtained very good sulfur removals but the reagents that are used are too expensive. Use of cheaper reagents leads to a loss of desired coal properties.

This study investigates the application of phase transfer catalysts to the selective oxidation of sulfur in coal using air and oxygen as oxidants. The phase transfer catalyst is expected to function as a selectivity moderator by permitting the use of milder reaction conditions than otherwise necessary. This would enhance the sulfur selectivity and help retain the heating value of the coal.

The use of certain coal combustion wastes for desulfurization, and the application of cerium (IV) catalyzed air oxidations for selective sulfur oxidation are also being studied. If successful this project could lead to the rapid development of a commercially viable desulfurization process. This would significantly improve the marketability of Illinois coal.

During this quarter aliquots of the IBC-101 coal have been ground to various particles sizes in an attempt to find the optimum physical pretreatment for mineral, especially pyrite, removal. Analysis of these various aliquots shows them to be representative of the original coal. In addition, preliminary desulfurization reactions using fly ash and scrubber sludges have been performed on an unoxidized IBC-101 sample. Results will be available next quarter. Also, SEM-EDAX analysis of the fly ash indicates that it contains oxides that have shown activity in base desulfurization reactions.

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EXECUTIVE SUMMARY

Due to environmental problems related to the combustion of high sulfur Illinois coal, there continues to be interest in the development of commercially viable pre-combustion desulfurization processes. These processes would allow the continued use of high sulfur Illinois coal for electricity generation, without the need for post combustion scrubbers.

Recent studies by the authors use a combination of a selective oxidation pretreatment reaction with a subsequent base desulfurization reaction to obtain very good sulfur removals (up to 95%). Unfortunately the cost of the chemical reagents used in the oxidative pretreatment is too high for commercial development and the use of cheaper oxidants results in too much carbon oxidation and hence Btu loss.

Although these studies failed to provide a cost effective desulfurization process, they do point us towards the types of chemical reactions needed for successful desulfurization. It is believed that the key to the success of the two-step process lies in the ability of the oxidant to selectively oxidize the organic sulfur species to their sulfoxides, sulfones or sulfonic acids in the pretreatment step. This selectively weakens the C-S bonds and therefore makes the subsequent removal of sulfur much easier.

The challenge that we face at this time is finding an inexpensive oxidant system that retains the desired sulfur selectivity and preserves the desired properties of the coal such as Btu value. Identifying this inexpensive oxidant system is the primary goal of this study. Specifically the application of phase transfer catalysts to the selective oxidation of sulfur over that of carbon, using air and oxygen as oxidants, is being investigated.

Phase transfer catalysts are substances that have the ability to transport chemical reagents across phase boundaries. This ability promotes chemical reactions in heterogeneous systems that would not otherwise take place or which would take place only very slowly. In this sense they are true catalysts and as such permit the formation of desired products under much milder, and therefore more selective, reaction conditions. It is these features that make phase transfer catalysts so attractive for reactions involving coal. It is anticipated that phase transfer catalysts would act as selectivity moderators in the oxidation of sulfur in coal.

Although the moderation of oxidative selectivity using phase transfer catalysts is the primary area for research in this project, a number of related features are also being examined. These include the potential use of coal combustion residues for the desulfurization step and the application of cerium (IV) catalyzed air oxidation to the selective oxidation of sulfur in coal.

Specific objectives have been identified for the completion of this project. These are:

1. Use physically cleaned coals from the Illinois Basin, to prepare a variety of selectively oxidized coals using air and oxygen under phase transfer catalysis conditions.

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2. Test these selectively oxidized coals for enhanced desulfurization under standard desulfurization conditions.
3. Compare these desulfurization results with those obtained for oxidized coals prepared without the phase transfer catalyst.
4. Investigate the applicability of cerium (IV) catalyzed air oxidation for selective oxidation of sulfur in coal, both with and without phase transfer catalysts.
5. Examine the ability of alkaline coal combustion wastes (scrubber wastes and fly ash) to desulfurize selectively oxidized and unoxidized coals.
6. Investigate the kinetics and mechanism of desulfurization under these various conditions by varying the time and temperature of the treatments on coals and model compounds.
7. Measure Btu contents after both oxidation and desulfurization reactions to quantify Btu recoveries after each process.
8. Test selected products for trace element content to determine if these elements can also be removed by the process.
9. Investigate the influence of the amount of phase transfer catalyst on the level of selective oxidation and subsequent desulfurization.
10. Examine the recovery of phase transfer catalysts from the reaction media to see if recycling of the catalyst would be possible.

As far as the authors have been able to determine the application of phase transfer catalysts to the selective oxidation of sulfur in coal has not been investigated before. Similarly, the authors could not find reference to the application of the cerium (IV) catalyzed selective air oxidation of organic sulfur to the study of coal desulfurization. In addition, the use of coal combustion wastes for the precombustion desulfurization of coals appears to be another unique feature of this project.

During the first quarter a number of achievements were made. These are:

- All coals, reagents, solvents, catalysts, fly ash and scrubber sludges have been obtained from their respective suppliers.
- A graduate student has been hired and trained in the following areas:
 - a) oxidation equipment
 - b) microreactor set-ups
 - c) fluidized sand baths
 - d) thermogravimetric analyzer
 - e) particle size analyzer
 - f) general laboratory procedures
 - g) laboratory safety

- An oxygen/air delivery system has been assembled and calibrated for the accurate and reliable supply of these gases during the oxidation reactions.
- The IBC-101 coal has been riffled into suitable aliquots and subjected to various physical pretreatments for the liberation of pyrite and other mineral matter. These processes include multiple pass micronization, liquid nitrogen treatment followed by multiple pass micronization, and sink/float treatment at 1.6 g/cm³.
- Preliminary desulfurization experiments using fly ash and scrubber sludge on an unoxidized IBC-101 coal have been performed.
- Many samples have been analyzed or submitted for analysis using TGA, the elemental analyzer, the Btu analyzer and the particle size analyzer. Many results have been obtained.
- Samples of the fly ash and scrubber sludge have been submitted for SEM-EDAX analysis to determine bulk chemical composition. Results for the fly ash have been obtained.

Summarizing the results this quarter the following conclusions can be made. Aliquots of the IBC-101 coal have been ground to various particle sizes in an attempt to find the optimum physical pretreatment for mineral, especially pyrite, removal. Analysis of these various aliquots shows them to be representative of the original coal. Preliminary desulfurization reactions using fly ash and scrubber sludges have been performed on an unoxidized IBC-101 sample. Results will be available next quarter. SEM-EDAX analysis of the fly ash indicates it contains oxides that have shown activity in base desulfurization reactions.

OBJECTIVES

The overall objective of this study is to develop a cost-effective process for the removal of organic sulfur from high sulfur Illinois coals. This project focuses on the use of phase transfer catalysts to moderate the selective oxidation of organic sulfur in coal using air and oxygen. The desulfurization of coal treated this way would be significantly enhanced. In addition, the use of coal combustion wastes to aid the desulfurization of these selectively oxidized coals is being explored.

A number of specific goals and objectives can be identified for the successful completion of this project. These are:

1. Use physically cleaned coals from the Illinois Basin to prepare a variety of selectively oxidized coals using air and oxygen under phase transfer catalysis conditions.
2. Test these selectively oxidized coals for enhanced desulfurization under standard desulfurization conditions.
3. Compare these desulfurization results with those obtained for oxidized coals prepared without the phase transfer catalyst.
4. Investigate the applicability of cerium (IV) catalyzed air oxidation for selective oxidation of sulfur in coal, both with and without phase transfer catalysts.
5. Examine the ability of alkaline coal combustion wastes (scrubber wastes and fly ash) to desulfurize selectively oxidized and unoxidized coals.
6. Investigate the kinetics and mechanism of desulfurization under these various conditions by varying the time and temperature of the treatments on coals and model compounds.
7. Measure Btu contents after both oxidation and desulfurization reactions to quantify Btu recoveries after each process.
8. Test selected products for trace element content to determine if these elements can also be removed by the process.
9. Investigate the influence of the amount of phase transfer catalyst on the level of selective oxidation and subsequent desulfurization.
10. Examine the recovery of phase transfer catalysts from the reaction media to see if recycling of the catalyst would be possible.

INTRODUCTION AND BACKGROUND

To reduce coal derived sulfur oxide emissions the electricity generating industry has been forced to switch to low sulfur coal and to use flue gas scrubbers. This has led to high unemployment in areas where high sulfur coal is mined and to the generation of significant quantities of scrubber wastes.(1)

Due to these considerations there continues to be interest in the development of pre-combustion desulfurization processes which would allow the continued use of high sulfur coal for electricity generation without the need for post combustion scrubbers.

Although some success has been achieved with the development of processes that can remove inorganic sulfur species from coal, an acceptable process for the removal of organic sulfur has yet to be found.

Laboratory studies have shown that very high levels of organic sulfur removal are technically possible. The problem has been that all of these processes have proven too expensive to become commercially viable.

Recent studies by the researchers used a combination of a selective oxidation pretreatment reaction with a subsequent base desulfurization reaction. Very good sulfur removals (up to 95%) were obtained but as with previous processes the cost of the reagents, especially those used in the selective oxidation step, inhibits the development of a commercial process (2-7). Use of cheaper oxidants were found to give similar levels of desulfurization but there are concerns regarding the selectivity of the oxidants and the possibility that too much carbon oxidation and therefore Btu loss occurs. The success of bases to desulfurize the oxidized coals raises the possibility that certain alkaline coal combustion wastes might be useful for desulfurization. If coal combustion wastes could be used for the desulfurization of coal, not only would process economics be influenced favorably, but a use for these problem wastes would be found. The possibility of using these waste materials for the desulfurization of coal is being investigated in this project.

Using XANES analysis it was found that oxidation with peroxyacetic acid converts most of the pyrite to sulfate and most of the sulfides and thiophenes to sulfoxides, sulfones and sulfonic acids.(8) This is the expected result. In addition the desulfurization treatment effectively removed the sulfoxides, sulfones and sulfonic acids that were formed by the oxidative pretreatment. Indeed, the only significant sulfur form still present after the combined treatment is thiophenic sulfur, and this has been reduced by some 70%. Both pyrite and organic sulfides were completely removed.

Although these studies failed to provide a cost effective desulfurization process, they do at least point us towards the kinds of processes that might become viable. It is believed that the key to the success of this process lies in the ability to selectively oxidize the organic sulfur

species to their sulfoxides, sulfones or sulfonic acids in the pretreatment step. This selectively weakens the C-S bonds and therefore makes the subsequent removal of sulfur much easier.

The problem that we face at this time is finding an inexpensive oxidant system that retains the desired sulfur selectivity. Unfortunately the inexpensive oxidants such as air, oxygen, nitric acid, etc. are not known for their oxidative selectivity and excessive oxidation of carbon is often observed.

For reasons that will be explained later it is believed that the selective oxidation of sulfur over carbon can be performed using inexpensive oxidants by employing substances called phase transfer catalysts (PTC). It is anticipated that the PTC would act as a selectivity moderator in the oxidation of sulfur in coal.

Phase transfer catalysts are substances that have the ability to transport chemical reagents across phase boundaries. This ability promotes chemical reactions in heterogeneous systems that would not otherwise take place or which would take place only very slowly. In this sense they are true catalysts and as such permit the formation of desired products under much milder, and therefore more selective, reaction conditions. It is these features that make phase transfer catalysts so attractive for reactions involving coal.

Phase transfer catalysts such as tetrabutyl ammonium hydroxide (TBAH) have been found to aid the swelling of the coal structure thereby allowing the penetration of other reactants. Indeed, the rate of O-methylation was not limited by the mass transport of the chemical reagents into the coal structure. This result supports the proposed mechanism whereby the PTC promotes the penetration of reagents into the coal matrix.

It is believed that phase transfer catalysts could be used to enhance the accessibility of oxidants in coal thereby permitting the use of mild oxidation conditions which would only oxidize sulfur. A highly selective reaction should be the result because, in most situations, the divalent sulfur atom is inherently more reactive towards oxidants than the carbon atom.(9,10) This is because the divalent sulfur atom can easily accommodate oxygen atoms becoming tetravalent or hexavalent without having to break any bonds. Carbon on the other hand is usually unable to form bonds with the oxygen without first breaking a C-C or C-H bond. This is why the oxidation of sulfur containing model compounds usually results in the formation of the sulfoxides, sulfones or sulfonic acids in high yield with no or very little oxidation of the carbon in the molecules.

The ability of PTCs to promote oxidations with air and oxygen from the gas phase has been demonstrated with the development of a number of synthetic pathways for organic chemicals. (11-14) For instance, fluorenone can be formed from fluorene using air as the oxidant and tricaprilylammonium chloride as a PTC. (11) Similarly 9,10 dihydroanthracene can be oxidized to anthraquinone by air with dicyldiethylammonium chloride as the PTC. (12) In both these

reactions the molar ratio of PTC to substrate was only 0.03 and the air pressure was 1 atm.

In addition, a new selective oxidation process has recently been developed for the conversion of sulfides into sulfoxides using air as the oxidant. This process uses a cerium catalyst $[(\text{NH}_4)_2\text{Ce}(\text{NO}_3)_6]$ to promote a single electron transfer from the sulfur species to molecular oxygen forming the radical cation and the superoxide anion, followed by triplet oxygen trapping of the radical cation. This catalytic air oxidation is also being investigated for the selective oxidation of sulfur in coal under PTC conditions.

EXPERIMENTAL PROCEDURES

The work that has been and will be undertaken during this project is outlined in the following text as a number of tasks.

Task 1. Sample selection and preparation

IBCSP coal samples Nos. 101 and 106 were selected for use in this study. These coals will be used because they have relatively high, but different, organic sulfur contents, and therefore provide the best challenge to the desulfurization process.

Each coal was riffled into suitable aliquot sizes and excessive deterioration prevented by storage under nitrogen. To provide beneficiated coal fines samples, aliquots of each coal was micronized using a nitrogen gas powered micronizer in order to liberate the very fine minerals found in these coals. Since previous results imply that pyrite removal prior to oxidation and desulfurization is essential, extra effort is being applied towards pyrite removal. To this end aliquots of coal have been passed through the micronizer up to three times while other aliquots have been treated with liquid nitrogen prior to micronization. The micronized coals are in the process of being centrifuged in a 1.6 g/cm^3 cesium chloride solution to establish the extent of pyrite liberation. After centrifugation the mineral-attenuated float material are washed with 1N HCl and distilled water, and finally dried under vacuum below 40°C . Coals treated in this way typically have less than 1% ash and very little residual pyrite. Analysis of these various products will determine the extent of physical pretreatment necessary to obtain optimum pyrite liberation. This level of coal preparation is required to provide a physically clean coal of very high quality. This will enable the desulfurization study to focus on organic sulfur removal, without worrying about any complications resulting from pyrite reactions or interactions.

Aliquots of each coal, both before and after physical cleaning, have been submitted for analysis including proximate, ultimate, Btu and particle size determination.

All solvents, reagents and catalysts have been obtained from chemical supply houses. Coal combustion wastes have been obtained from the Coal Combustion Residues Management (CCRM) Program Sample Bank maintained by the mining engineering department at SIUC.

Task 2. Selective oxidation using phase transfer catalysts.

Two phase transfer catalysts are being examined in this study. These are tetrabutylammonium hydroxide (TBAH) and tetrabutylammonium chloride (TBAC). These PTC's were chosen because they have been used in coal related studies before and because one is a base while the other is a neutral salt. Comparison of the two will therefore help to establish the role of pH in these oxidations.

Three oxidant systems are to be evaluated under PTC conditions. These are air oxidation, oxygen oxidation and cerium (IV) catalyzed air oxidation. Each will be studied in the presence of each PTC and also in the absence of any PTC. Four temperatures with a standard reaction time of 1 hour will be investigated for each PTC/oxidant combination. This will give the reaction matrix shown in Table 2.

Table 2. Reaction matrix for PTC assisted oxidations

PTC/Oxidant Combination	0°C	20°C	50°C	100°C
no oxidant	X	X	X	X
air	X	X	X	X
oxygen	X	X	X	X
cerium/air	X	X	X	X
air/TBAH	X	X	X	X
air/TBAC	X	X	X	X
oxygen/TBAH	X	X	X	X
oxygen/TBAC	X	X	X	X
cerium/air/TBAH	X	X	X	X
cerium/air/TBAC	X	X	X	X

For these initial investigations the oxidant flow rate would be held constant at 20 ml/min and the PTC concentration maintained at 5% (wt% based on coal). Each experiment will be conducted on 5 grams of coal. This will provide sufficient sample for the subsequent desulfurization reaction, and for the Btu and proximate analysis of products both after oxidation and again after desulfurization.

Task 3. Optimization of pretreatment reaction

At this point the most promising oxidative pretreatment process will be selected for further detailed study. This study will investigate variations in PTC concentration, other oxidant flow rates and different reaction times and temperatures. Many of these variables would be determined from an analysis of the results obtained in Task 2. This new data will be used to help identify the oxidation conditions that lead to optimized levels of sulfur removal with subsequent desulfurization reactions. In addition, the recovery of the phase transfer catalysts from the reaction medium would be examined under this task.

Task 4. Standard desulfurization reaction

Each selectively oxidized coal and unoxidized control coal would be desulfurized under standard desulfurization conditions. A typical desulfurization experiment involves dispersing 2 grams of coal (oxidized or unoxidized) in 4 mL of methanol with 1 gram of sodium hydroxide. This slurry is placed in a 10 mL stainless steel microreactor. The microreactor is purged with argon, sealed and then placed in a fluidized sand bath maintained at a temperature of 350°C for 60 minutes. After this time the reactor is cooled to room temperature and the contents collected at the filter. The product is washed with dil. HCl then distilled water and finally dried overnight in a vacuum oven. Products are then be submitted for analysis as described in Task 7.

Task 5. Desulfurization with coal combustion residues

To test the ability of scrubber sludge and fly ash for the desulfurization of coal, up to five selectively oxidized samples and the unoxidized sample from each coal would be selected. The desulfurization reaction would use the same conditions as used for the standard desulfurization procedure but the sodium hydroxide would be replaced with an equal quantity of the combustion waste. Products would be tested as described in Task 7.

Task 6. Studies involving sulfur model compounds

A set of sulfur-containing model compounds comprising a thiol, an aliphatic sulfide, an alkyl aryl sulfide, an aryl aryl sulfide, an aliphatic disulfide, an aromatic disulfide, a thiophene, benzothiophene and dibenzothiophene will be oxidized using the conditions identified in Task 2. The oxidation products (probably sulfonic acids and sulfones) will be characterized and then treated using the standard desulfurization conditions. Desulfurization of this model compound mixture would also be attempted using the coal combustion waste identified in Task 5. GCMS and GC-FID/FPD analysis of the products will be used to identify any desulfurized model compound products. Compounds identified in this way will be used in attempts to elucidate the mechanism of desulfurization and as a characterization tool for the organic sulfur in coal.

Task 7. Product analysis and evaluation

The analysis of all products obtained during this work will be performed. Initially, this will involve recording the product yield, the sulfur content and the ash/moisture content. Other tests such as proximate analysis, Btu analysis, elemental analysis and sulfur forms analysis will be performed throughout the project on selected samples. In addition selected products will be examined by digestion and then ICP-ES analysis to monitor trace element concentrations. The information provided will be used to evaluate the processes under investigation and to indicate the direction of future experiments.

RESULTS AND DISCUSSION

During the first quarter a number of achievements have been made. These are listed below:

- All coals, reagents, solvents, catalysts, fly ash and scrubber sludges have been obtained from their respective suppliers.
- A graduate student has been hired and trained in the following areas:
 - a) oxidation equipment
 - b) microreactor set-ups
 - c) fluidized sand baths
 - d) thermogravimetric analyzer
 - e) particle size analyzer
 - f) general laboratory procedures
 - g) laboratory safety
- An oxygen/air delivery system has been assembled and calibrated for the accurate and reliable supply of these gases during the oxidation reactions.
- The IBC-101 coal has been riffled into suitable aliquots and subjected to various physical pretreatments for the liberation of pyrite and other mineral matter. These processes include multiple pass micronization, liquid nitrogen treatment followed by multiple pass micronization, and sink/float treatment at 1.6 g/cm^3 .
- Preliminary desulfurization experiments using fly ash and scrubber sludge on an un-oxidized IBC-101 coal have been performed.
- Many samples have been analyzed or submitted for analysis using TGA, the elemental analyzer, the Btu analyzer and the particle size analyzer. Many result have been obtained.

- Samples of the fly ash and scrubber sludge have been submitted for SEM-EDAX analysis to determine bulk chemical composition. Results for the fly ash have been obtained.

Sample Preparation

Available data for the physically cleaned coals are shown in Table 2.

Table 2.

Coal	pretreatment	micron passes	size μ	C	H	N	S	Ash	Moi.	Btu
101	none	0	N/A	69.8	4.44	1.17	4.38	10.3	11.4	12596
101	none	1	9.9	66.8	4.26	1.02	4.52	12.4	5.87	12294
101	none	2	6.76	66.0	4.62	1.11	4.46	12.8	3.84	12144
101	none	3	4.49	65.3	4.22	0.99	4.39	12.6	3.31	12122
101	cryo	0	N/A	70.4	3.94	1.15	4.32	9.61	12.2	12453
101	cryo	1	11.8	69.9	4.1	1.06	4.35	10.8	8.12	12292
101	cryo	2	6.43	67.9	4.32	0.99	4.29	10.9	3.68	12136
101	cryo	3	4.85	66.8	4.23	0.92	4.33	10.8	3.64	12132

As expected there is increased particle size reduction with increasing passes through the micronizer. This is true both with and without the cryogenic pretreatment. It is surprising however, that the particle sizes for the cryogenically treated sample are larger than those produced without the freeze-thaw cycle provided by the addition of the liquid nitrogen. It was expected that the cracks resulting from the cryogenic pretreatment would have led to smaller particle sizes upon subsequent micronization. The fact that the cryogenic pretreatment followed by micronization does not produce smaller particle sizes than micronization alone does not mean that the liquid nitrogen treatment failed to give enhanced pyrite/mineral liberation. This information will be provided by the sink/float tests.

The Btu values decline slightly with increased grinding indicating that there is some oxidation during repeated micronization. However, the change in the Btu value is small and the levels of oxidation are not considered sufficient to influence subsequent reactions.

In general the elemental, proximate and Btu data agree very well with that provided by the Illinois Basin Coal Sample Program. It can be seen that the micronizer has a drying effect in that the moisture contents decline on repeated passes through the micronizer. As expected

there are no major differences in the elemental composition of the various aliquots of the IBC-101 coal. This indicates that the various physical pretreatments have not resulted in the fractionation of the sample and that each aliquot is a true representation of the original coal.

Fly ash and Scrubber Sludge Desulfurizations

The preliminary desulfurizations using fly ash and scrubber sludge that have been performed in the previous quarter are listed in Table 3. At this time only standard conditions of 350°C for 60 minutes have been examined. Other conditions may be used in the future if the results suggest this is necessary.

Table 3. Fly ash and scrubber sludge desulfurization conditions

Coal	Solvent	Reagent	Time	Temp
101	water	none	60 mins	350°C
101	methanol	none	60 mins	350°C
101	water	NaOH	60 mins	350°C
101	methanol	NaOH	60 mins	350°C
101	water	Fly Ash	60 mins	350°C
101	methanol	Fly Ash	60 mins	350°C
101	water	Scrubber S.	60 mins	350°C
101	methanol	Scrubber S.	60 mins	350°C

The products from these reactions are currently being worked-up. Results will be available for the next quarterly report.

SEM-EDAX analysis of fly ash

Analysis of the fluorescent X-rays produced by the defocussed electron beam in the SEM was used to establish the bulk chemistry of the fly ash in terms of the oxide content. Based upon previous determinations of this type Si, Al, Fe, Na, Ca, K, Ti, Mg, S and Mn were determined as their oxides. The results are given in table 4.

Bulk chemical analysis of the fly ash using semi-quantitative SEM-EDAX indicates that there are oxides of sodium, potassium and calcium, among others, all of which may take part in base desulfurization reactions. Of course the SEM-EDAX results are reported as oxide contents and these elements may not necessarily be in the oxide form. However, a water

slurry of the fly ash is strongly alkaline with a pH of around 12-13., suggesting oxides are present.

Similar tests of the scrubber sludge are underway.

Table 4. Bulk chemical composition of fly ash determined by SEM-EDAX.

Element	Wt%	Formula	Oxide %
Na	0.86	Na ₂ O	1.16
Mg	0.26	MgO	0.43
Al	10.32	Al ₂ O ₃	19.50
Si	23.86	SiO ₂	51.04
S	1.18	SO ₃	2.94
K	1.86	K ₂ O	2.24
Ca	3.17	CaO	4.44
Ti	0.84	TiO ₂	1.39
Fe	11.79	Fe ₂ O ₃	16.85
O	45.87		

CONCLUSIONS AND RECOMMENDATIONS

Aliquots of the IBC-101 coal have been ground to various particles sizes in an attempt to find the optimum physical pretreatment for mineral, especially pyrite, removal. Analysis of these various aliquots shows them to be representative of the original coal.

Preliminary desulfurization reactions using fly ash and scrubber sludges have been performed on an unoxidized IBC-101 sample. Results will be available next quarter.

SEM-EDAX analysis of the fly ash indicates its contains oxides that have shown activity in base desulfurization reactions.

DISCLAIMER STATEMENT

This report was prepared by Dr. Stephen R. Palmer, SIUC with support, in part by grants made possible by the U.S. Department of Energy Cooperative Agreement Number DE-FC22-92PC92521 (Year 4) and the Illinois Department of Commerce and Community Affairs through the Illinois Coal Development Board and the Illinois Clean Coal Institute. Neither

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PROJECT MANAGEMENT REPORT
September 1, 1995 through November 30, 1995

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Processes, Southern Illinois University at
Carbondale
Project Manager: Dr. Ken Ho, Illinois Clean Coal Institute

COMMENTS

None.

DISCLAIMER

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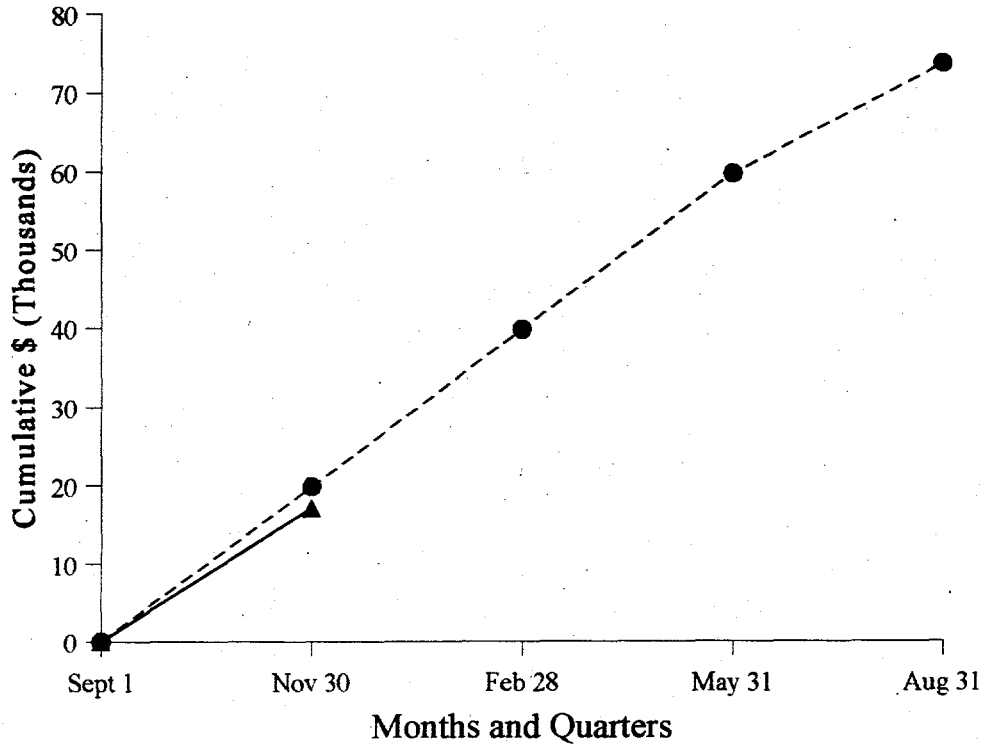
PROJECTED AND ESTIMATED EXPENDITURES BY QUARTER

Quarter*	Types of Cost	Direct Labor	Fringe Benefits	Materials and Supplies	Travel	Major Equipment	Other Direct Costs	Indirect Cost	Total
Sept. 1, 1995 to Nov. 30, 1995	Projected	10,855	3,010	1,875	0	0	2,375	1,812	19,927
	Estimated	12,760	1,833	394	0	0	619	1,561	17,167
Sept. 1, 1995 to Feb. 28, 1996	Projected	21,710	6,020	3,750	0	0	4,750	3,623	39,853
	Estimated								
Sept. 1, 1995 to May 31, 1996	Projected	32,565	9,030	5,625	0	0	7,125	5,435	59,780
	Estimated								
Sept. 1, 1995 to Aug. 31, 1996	Projected	39,879	11,218	7,500	500	0	8,000	6,710	73,807
	Estimated								

*Cumulative by Quarter

CUMULATIVE COSTS BY QUARTER

Desulfurization of Coal: Enhanced Selectivity Using Phase Transfer Catalysts

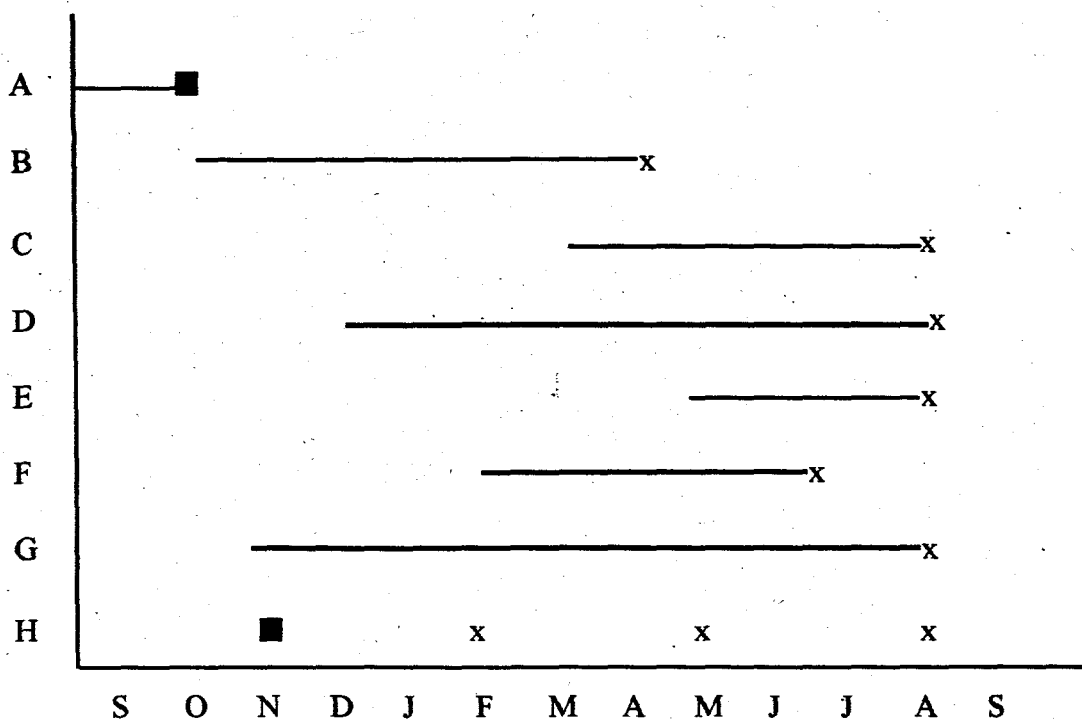


● = Projected Expenditures - - - - -

▲ = Actual Expenditures _____

Total Illinois Clean Coal Institute Award \$73,807

SCHEDULE OF PROJECT MILESTONES



Begin
Sept. 1
1995

Hypothetical Milestones:

- A: Sample preparation
- B: Selective oxidation with PTC
- C: Optimization
- D: Standard desulfurization reaction
- E: Desulfurization with coal wastes
- F: Model compounds
- G: Product evaluation
- H: Reporting

Comments:

None.