

DOE/PC/92521--T275

TECHNICAL REPORT

September 1, 1995, through November 30, 1995

Project Title: **DESULFURIZATION OF ILLINOIS COALS WITH  
HYDROPEROXIDES OF VEGETABLE OILS AND ALKALI**

DOE Cooperative Agreement Number: DE-FC22-92PC92521 (Year 4)  
ICCI Project Number: 95-1/1.1D-2P  
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ABSTRACT

Organic sulfur is removed from coals by treatment with aqueous base, air, and vegetable oils with minimal loss of BTU. Such results were revealed during exploratory experiments on an ICCI funded project to remove organic sulfur from Illinois coals with hydroperoxides of vegetable oils. In fact, prewashing IBC-108 coal with dilute alkali prior to treating with linseed oil and air results in 26% removal of sulfur. This new method will be investigated by treating coals with alkali, impregnating coals with polyunsaturated oils, converting the oils to their hydroperoxides, and heating. Since these oils are relatively inexpensive and easily applied, this project could lead to a cost effective method for removing organic sulfur from coals. Moreover the oils are environmentally safe; they will produce no noxious products and will improve burning qualities of the solid products. During this first quarter the selection of base for pretreatment and extraction (Task 1) has been completed. NaOH is better than  $\text{NH}_4\text{OH}$  for the pretreatment and  $\text{Na}_2\text{CO}_3$  is better than NaOH for the oil extraction. About 40% of sulfur is removed from IBC-108 coal using 5% NaOH for pretreatment followed by linseed oil oxidation in air and  $\text{Na}_2\text{CO}_3$  extraction.

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## EXECUTIVE SUMMARY

This project proposes to remove organic sulfur from coal, maintain its BTU, and increase its volatiles, by a new process of pretreating with alkali and impregnating with polyunsaturated vegetable oils. Catalyzed by coal, air converts these oils into their hydroperoxides which are powerful oxidizing agents. A similar agent, peroxyacetic acid, has shown in a previously funded ICCI project to desulfurize coal. But polyunsaturated vegetable oils have advantages which make them attractive for treating coal. First, these compounds are inexpensive, renewable natural products available from Illinois farms; second, they possess chemical properties which can be directed toward oxidizing organic sulfur; third, they furnish carbonaceous residues which will increase BTU's and volatiles; and fourth, they are environmentally safe and produce no noxious products.

Preliminary experiments at SIUC have shown that when IBC-108 coal is impregnated with linseed oil and heated in air at 50-100°C, its organic sulfur is removed. The results showed that coal catalyzes formation of hydroperoxides in the oil and these hydroperoxides oxidize the organic sulfur with minimum loss of BTU. Additionally, experiments have shown that pretreating IBC-108 coal with NaOH increases the amount of organic sulfur removed during subsequent treatment with linseed oil. This project proposes to build on this new evidence and to aim at a technically feasible and economically viable process step.

Three tasks are proposed: **Task 1** will select the bases for pretreating and extraction. Tests will examine NaOH and  $\text{NH}_4\text{OH}$  at two different concentrations followed by treatment with linseed oil at 100 °C for 20 hours, then extraction with two different bases, NaOH and  $\text{Na}_2\text{CO}_3$ . **Task 2** will determine the abilities of the above selected bases in combination with linseed oil to remove organic sulfur from IBC-108 coal. Tests of base pretreatment will be conducted at 25°C, two different concentrations, and four different times. Tests of oil treatment will be conducted at two different oil:coal ratios, three different reaction times, three different temperatures, and with two different extraction solutions. This task will produce 144 experiments with accompanying plots of sulfur removal as functions of temperature and time and will be used to study reaction rates and the mechanism(s) of sulfur removal. **Task 3** will determine the mass balance along with the volatiles and BTU changes from the experiments in Tasks 1 and 2 creating a data base of 152 analyses of mass balances, volatiles, and BTU's along with plots of their dependencies on temperature and time. All of these data will be examined for clues to the mechanism(s) of organic sulfur oxidation and removal from Illinois coals.

During this first quarter we completed screening of the bases for pretreating the coal and for extracting the oil after oil treatment. We found that pretreating IBC-108 coal with bases removes 13% to 23% of the sulfur. NaOH is a better pretreatment than  $\text{NH}_4\text{OH}$  in most experiments and  $\text{Na}_2\text{CO}_3$  is better than NaOH for the final extraction. Higher temperatures and higher base concentrations remove more sulfur. Thereby, treatments with 5%  $\text{NH}_4\text{OH}$  at 100°C in the presence of bubbling  $\text{O}_2$  or with 5% NaOH at 25°C remove more sulfur (21-23%) than any other treatment with alkali alone. However, even more

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sulfur is removed from the pretreated coal by linseed oil treatment followed by base extraction. And the best results (about 40% sulfur removal) are obtained with the combinations of 5% NaOH-OIL-5% Na<sub>2</sub>CO<sub>3</sub> (B-O-C), 1% NH<sub>4</sub>OH-OIL-5% Na<sub>2</sub>CO<sub>3</sub> (NI-O-C), and 5% NH<sub>4</sub>OH (100°C)-OIL-5% Na<sub>2</sub>CO<sub>3</sub> (N<sub>100</sub>-O-C). This base-oil-base treatment appears to selectively remove organic sulfur and it does this with minimum loss of BTU.

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## OBJECTIVES

The goal of this project is to develop a cost effective method to remove organic sulfur from pyrite-free and mineral-free coal. The objective is to test the feasibility of using alkali and inexpensive, renewable farm products to desulfurize Illinois coals. The specific objectives of this project are:

1. determine the ability of alkali and oils to remove organic sulfur from Illinois coals,
2. establish the volatile and BTU changes from treating Illinois coals with alkali and oils,
3. establish the mass balance of solids, liquids, and gases resulting from treating Illinois coals with alkali and oils, and
4. study the reaction rate(s) and mechanism(s) of sulfur removal from Illinois coals treated with alkali and oils.

Briefly the tasks scheduled for the current year are:

**Task 1.** *Selection of base for pretreatment and extraction*

**Task 2.** *Determination of base and linseed oil ability to remove organic sulfur from IBC-108 coal*

**Task 3.** *Determination of mass balance, volatiles, and BTU*

## INTRODUCTION AND BACKGROUND

### **Relevance to Illinois basin coal problem and Unique Aspects**

This project is relevant to solving the problem of high sulfur content of Illinois coals. Its significance rests on its use of inexpensive farm materials to remove organic sulfur in Illinois coals.

The potential importance of this project is its impact on the marketability of Illinois coals. Producing clean products from coal will add to the economic importance of coal. This project has the potential of utilizing cheap, renewable farm products for enhancing coal conversion process, especially for removing sulfur and thereby upgrading solid products.

The unique aspect of this project is its use of inexpensive farm products, such as linseed oil (\$0.28/LB) and other vegetable oils, to achieve desulfurization and upgrade char. These farm products are cheap enough that they need not be recycled, rather, they enrich the coal conversion products. On an equivalent weight basis (gram molecular weight per

hydroperoxy group), linseed oil with maximum hydroperoxy groups contains about 86% of the oxidizing ability of peroxyacetic acid. Yet the cost of each hydroperoxy group in linseed oil is only 23% of the cost of each hydroperoxy group in peracetic acid. Therefore this project has the unique aspects not only of being environmentally safe, disrupting the coal matrix, increasing volatiles, oxidizing the sulfur, and adding carbon, oxygen, and hydrogen to the char, but also of being less expensive than peroxyacetic acid.

### **Background**

Each year Illinois farms produce millions of tons of usable and unusable materials which are easily collected, easily transported, and readily available near Illinois coal mining districts. These materials consist mainly of carbohydrates, fats, and oils, which contain hydrogen and oxygen, elements in low concentrations in coals. Therefore, these materials are potentially valuable for coal conversion processes.

There are good reasons for exploring carbohydrates, fats, and oils as participants in coal conversion reactions. First, these compounds are inexpensive and renewable natural products available from Illinois farms; second, they possess chemical properties which can be directed toward oxidizing organic sulfur; third, they furnish carbonaceous residues which will increase BTU's of coal char; and fourth, they are environmentally safe and produce no noxious products.

Hydrogen in some form is frequently added during coal conversion processes. Besides adding hydrogen as  $H_2$  gas or as some readily dehydrogenatable molecule, such as tetralin, hydrogen has been added in the form of other hydrogen-rich organic molecules, such as ethanol and methane<sup>1-3</sup>. Other rich sources of hydrogen are fats and oils from vegetable and animal materials. Carbohydrates likewise contain hydrogen, although not as much on a molar basis as fats and oils. But, carbohydrates contain much more oxygen than fats and oils on a molar basis.

Using carbohydrates as well as fats and oils as sources of oxygen may be beneficial to pyrolysis and desulfurization because small amounts of oxygen seem to increase desulfurization. For example, ICCI funded coal treatments with methane/oxygen<sup>2</sup>, ethanol<sup>3</sup>, lignin<sup>4</sup>, a proprietary oxidant<sup>5</sup>, and air<sup>5,6</sup> are all processes in which oxygen, either added or present in the reactants, is beneficial to pyrolysis and desulfurization. Therefore, their oxygen contents make carbohydrates, fats, and oils likely candidates for enhancing coal conversion processes. Moreover, their oxygen may become incorporated into the products and increase their octane ratings. So using carbohydrates, fats, and oils makes chemical sense as sources of oxygen in coal conversion processes.

However, using carbohydrates, fats, and oils merely as sources of hydrogen, carbon, and oxygen is overlooking important coal desulfurization chemistry. For example, fats and oils contain labile allylic hydrogens which react with oxygen in air to form hydroperoxides. These hydroperoxides lead to rancidity, and some oils are so prone to this reaction that radical inhibitors are regularly added to preserve them for the food market. Thus oils can be

converted into powerful oxidizing agents by forming hydroperoxides, and these can oxidize organic and inorganic sulfur in coals.

Formation of hydroperoxides in oils occurs from the reaction of singlet oxygen at allylic positions on unsaturated fatty acids. But singlet oxygen is not ordinary oxygen. Singlet oxygen is the excited state of ground-state oxygen (a triplet) and is formed in very low concentrations in air by action of light. Singlet oxygen is not formed in high concentrations because triplet oxygen does not readily absorb energy from light. Thus the rate of formation of hydroperoxides is ordinarily slow in air.

However, the rate of formation can be greatly increased by increasing the concentration of singlet oxygen through photosensitization. Photosensitization methods employ a photosensitizer molecule which absorbs energy from light and transfers that energy to triplet oxygen, raising it into the excited singlet state. Photosensitizers need be present in only low concentrations, so one of the tasks in the 1994-5 project was to test standard photosensitizers and **coal as a sensitizer**.

Results have shown that coal is indeed a sensitizer, perhaps because it contains radicals<sup>7,8,9</sup>, which apparently directly convert triplet to singlet oxygen. During the first five months of the current funded project, we have found that coal alone amply catalyzes the formation of hydroperoxides in linseed oil. (We chose linseed oil for tests because it is readily available at the grocery store and relatively rich in polyunsaturated fats, such as glyceryl oleate, and glyceryl linoleate.) In fact, formation of hydroperoxides in the presence of coal equals that in the presence of a known photosensitizer and ultraviolet light. Apparently, coal is a very good singlet oxygen initiator.

We tested the ability of coal to sensitize oxygen in air and produce hydroperoxy groups in linseed oil because these hydroperoxy groups are powerful oxidizing agents. Similar powerful oxidizing agents, such as peroxyacetic acid, had been used in earlier ICCI funded projects to desulfurize coal<sup>10,11</sup>. Moreover, similar methods are well known for oxidizing organic sulfur to sulfate<sup>12-15</sup>. Since hydroperoxides possess about the same oxidizing ability as peracids, it is reasonable to investigate inexpensive hydroperoxides from vegetable oils for desulfurizing coals.

But does linseed oils actually desulfurize coals? To determine whether any coal desulfurization actually occurred we conducted the following experiments: A thin coat of linseed oil was placed on 5g of IBC-108 coal by making a slurry of the coal in a solution of the weighed oil in 30 mL of chloroform and then evaporating the chloroform. The oil-coal mixture was spread uniformly on a petri dish, which was floated on a constant temperature bath at either 100 °C or 50 °C with or without UV irradiation for 18hrs. The reaction mixture was extracted with 50 mL of tetrahydrofuran and chloroform, the coal was dried, and its sulfur content determined. Clearly, merely treating the coal with linseed oil removes some organic sulfur. Ultraviolet irradiation improves sulfur removal, but increasing the temperature removes more.



In order to remove the oil from the coals so sulfur analyses could be performed, we treated the reacted oil-coal mixture with NaOH. This hydrolyses the oil into the sodium salt of the fatty acids (soap) and glycerol, both of which are water soluble. During experimentation with various procedures, we discovered that pretreatment of the coal with NaOH resulted in subsequently better sulfur oxidation by the impregnated linseed oil. Clearly, pretreatment with NaOH influences the coal matrix such that it is more susceptible to oil impregnation and sulfur oxidation.

Bases, especially  $\text{NH}_4\text{OH}$ , are known to modify coal matrices by chemical comminution<sup>17</sup>. And  $\text{NH}_4\text{OH}$  has been used to pretreat coal before oxidation with NaOCl (sodium hypochlorite) followed by  $\text{Na}_2\text{CO}_3$  extraction in a procedure for removing organic sulfur from coals<sup>18</sup>. Therefore, this project proposes to test both NaOH and  $\text{NH}_4\text{OH}$  as pretreatments of IBC-108 coal followed by treatment with the hydroperoxides of linseed oil and extracted with NaOH or  $\text{Na}_2\text{CO}_3$ .

Linseed oil has been and will continue to be preferentially used in this project because it is inexpensive, can be easily sprayed on coal, and possesses a high degree of unsaturation. Its main unsaturated groupings are the linolenate group (approximately 58%) which contains three double bonds and the linoleate group (approximately 27%) which contains two double bonds. So approximately 85% of linseed oil is composed of these highly unsaturated groupings.

In summary, adding oils to coals offers:

1. ***in situ* formation of hydroperoxides**, which are powerful oxidizing agents that can oxidize organic sulfur and lead to coal detoxification,
2. **environmental safety of zero discharge**; the oil need not be removed but can remain with the coal, and no noxious products will be formed,
3. **increased volatiles**; the oil will produce volatiles which will enhance the burning qualities of the treated coal,
4. **increased hydrogen content**; the high hydrogen content of the oils will be available to the coal,
5. **increased BTU**; the oils furnish carbon and hydrogen which will increase the heat content of the coal,
6. **decreased costs**; in pure form and truck load quantities these oils can be purchased for \$0.28/LB; however, in raw form and tank car quantities the price will be much less. Eliminating purification steps necessary for current markets will reduce the cost of oils, and

7. **ease of use**; vegetable oils should be easily added to coals by simply spraying a thin film on finely divided coal.

## EXPERIMENTAL PROCEDURES

### Description of Work Proposed

#### **Task 1.** *Selection of base for pretreatment and extraction*

Screening of the base to use for pretreating the coal will be conducted in a systematic way: Eight 5 g portions (four pairs) for IBC-108 coal will be immersed for 10 hours at 25°C by pairs in one of the following aqueous solutions: 1% NaOH, 5% NaOH, 1% NH<sub>4</sub>OH, or 5% NH<sub>4</sub>OH. These eight pretreated portions of coal will each be washed with distilled water until the wash waters are neutral and then dried and treated with linseed oil for 20 hours at 100°C. One member of each pair will then be washed with a solution of 5% NaOH, followed by washings with water and organic solvents, and the other member of the pair will be washed with a solution of 5% Na<sub>2</sub>CO<sub>3</sub>, followed by washings with water and organic solvents. Sulfur analyses will be obtained on each sample before and after oil treatment, such that comparisons can be made between the samples treated with base only and the same samples receiving the full treatment. Based on which base produces the lowest sulfur content, one base, either NaOH or NH<sub>4</sub>OH, will be chosen for pretreating coal and one washing solution, either aqNaOH or aqNa<sub>2</sub>CO<sub>3</sub>, will be chosen for washing the oil-treated samples in the next task.

#### **Task 2.** *Determination of base and linseed oil ability to remove organic sulfur from IBC-108 coal*

We will test different methods of using linseed oil to remove sulfur from IBC-108 coal. This is the coal from the Illinois Basin Coal Sample Program maintained in Champaign by the Illinois State Geological Survey. Its descriptions are shown in Table 1. Best conditions for the full treatment will be narrowed by a series of 144 experiments in which 5 g portions of IBC-108 coal are treated variously with two concentrations of base at four different temperatures followed by treatment with linseed oil (two different ratios of oil:coal) at three different temperatures for three different times, and finally washed in either NaOH or Na<sub>2</sub>CO<sub>3</sub> (depending on which is chosen in Task1) and organic solvents. Mass balances will be obtained on all experiments; sulfur, volatiles, and BTU analyses will be obtained from the Department of Mechanical Engineering at SIUC.

**Table 1. Descriptions of IBC-108 Coals**

**IBC 108.** This is a micronized blend of Herrin and Springfield coals (80% and 20%, respectively) cleaned by an advanced froth flotation process (microbubble column flotation) in 1988. It is delivered to requesters as a filter cake (approximately 45% moisture). It is ideal for investigators wishing to use a deep-cleaned Illinois coal with low pyrite content.

**Coal analyses (% , moisture free basis except moisture).**

	<u>Avg.</u>	<u>SD</u>
Vol. Matter	41.6	0.57
Fixed Carbon	54.7	0.58
H-T Ash	3.7	0.19
Carbon	76.0	0.34
Hydrogen	5.2	0.31
Nitrogen	1.5	0.11
Oxygen	10.9	0.59
Total C	0.0	0.03
Total Sulfur	2.7	0.07
Sulfatic	0.0	0.03
Pyritic	0.4	0.07
Organic	2.3	0.09
TU/lb	13726	66.33
FSI	3.2	0.78

**Task 3. Determination of mass balance, volatiles, and BTU**

Task 3 will furnish information about how mass balance, volatiles, and BTU vary with the conditions in Task 2. This information will tell how much coal is extracted in the process and furnish a way to correct sulfur, volatiles and BTU analyses for changes in mass of the coal during the process. By comparing the rates of change of mass, sulfur contents, volatiles, and BTU it will be possible to evaluate the feasibility of the process.

**RESULTS AND DISCUSSION**

Testing for effectiveness of sulfur removal consisted of the following procedure: a thin layer of linseed oil was placed on the coal in a petri dish first by making a slurry of the coal in a solution of oil in hexane and then by evaporating the hexane. The oil-coal mixture on the petri dish was heated at the reaction temperature by floating it in a constant temperature bath for 20 hours. After reaction, the oil was treated with base to saponify the oil and to leach out some sulfur compounds from the coal matrices. The oil extraction with 5% NaOH was carried out at 25 °C for 24 hours and with 5% Na<sub>2</sub>CO<sub>3</sub> at 80 °C for 2 hours. After the base treatments, the coal samples were thoroughly washed with water and with 50 mL of methanol, then dried and submitted for sulfur and BTU analyses. The results are listed in Table 2.

In all experiments, chlorinated solvents, such as chloroform and tetrachloroethylene, were completely eliminated because of the possible interference of chlorine with sulfur analyses as suggested by project monitor.

**Table 2: Sulfur and BTU Analyses**

Exp. No.	Coal (g)	Treatment <sup>a</sup> (conditions)	Sulfur (%)	% Sulfur removed	BTU (lb.)
1	20	B (5% NaOH @ 25 °C)	2.04	22.7	11,800
2	20	BI (1% NaOH @ 25 °C)	2.14	18.9	12,251
3	20	N (5% NH <sub>4</sub> OH @ 25 °C)	2.19	17.0	12,905
4	20	NI (1% NH <sub>4</sub> OH @ 25 °C)	2.29	13.3	13,045
5	20	N <sub>100</sub> (5% NH <sub>4</sub> OH @ 100 °C)	2.08	21.2	12,047
6	20	N <sub>100/Ox</sub> (5% NH <sub>4</sub> OH @ 100 °C + O <sub>2</sub> )	2.04	22.7	11,985
7	5	B-O-B	1.80	31.8	11,809
8	5	B-O-C	1.60	39.4	12,089
9	5	N <sub>100/Ox</sub> -O-B	1.77	33.0	11,676
10	5	N <sub>100/Ox</sub> -O-C	1.66	37.1	11,835
11	5	N-O-B	1.92	27.3	11,825
12	5	N-O-C	1.79	32.2	12,258
13	5	BI-O-B	1.95	26.1	11,986
14	5	BI-O-C	1.79	32.2	12,369
15	5	NI-O-B	1.96	25.8	12,964
16	5	NI-O-C	1.61	39.0	13,125
17	5	N <sub>100</sub> -O-B	1.80	31.8	11,948
18	5	N <sub>100</sub> -O-C	1.58	40.2	12,136

<sup>a</sup> B: 5% NaOH @ 25 °C, BI: 1% NaOH @ 25 °C, N: 5% NH<sub>4</sub>OH @ 25 °C, NI: 1% NH<sub>4</sub>OH @ 25 °C, C: 5% Na<sub>2</sub>CO<sub>3</sub> @ 80 °C, N<sub>100</sub>: pretreated with 5% NH<sub>4</sub>OH @ 100°C, N<sub>100/Ox</sub>: pretreated with 5% NH<sub>4</sub>OH @ 100°C with O<sub>2</sub> bubbling.

Figure 1 shows results of pretreating IBC-108 coal with NaOH and NH<sub>4</sub>OH under different conditions. The sulfur removal ranges from 13.3% to 22.7%. Sulfur removal is favored by higher base concentrations and higher temperatures, and the presence of oxygen has little effect on sulfur removal (compare N<sub>100</sub> with N<sub>100/Ox</sub>). NaOH is more effective than NH<sub>4</sub>OH under the same conditions (compare B with N, and BI with NI).

Figures 2 and 3 show the effects on sulfur removal of oil treatment and base extractions after the oil treatment. Clearly, more sulfur is removed from the pretreated coals. After oil treatment, extraction with Na<sub>2</sub>CO<sub>3</sub> is more effective than extraction with NaOH. For example, pretreating with 5% NaOH alone removes 22.7% of the sulfur, pretreating with 5% NaOH, then linseed oil, and finally 5% NaOH (B-O-B) removes 31.8 % of the sulfur, and pretreating with 5% NaOH, then linseed oil, and finally 5% Na<sub>2</sub>CO<sub>3</sub> (B-O-C) removes 39.4% of the sulfur.

Figures 4-6 show BTU changes under various treatments. Pretreating with 5% NaOH causes loss of 11% BTU. However, pretreatment with base followed by first oil and then base again causes loss of only 10% (B-O-B) and 8% (B-O-C) BTU. Comparing these

results to those in Figures 1 to 3 suggests that the base-oil-base treatments remove organic sulfur but only slightly remove other organics which do not contain sulfur. Perhaps oxidation by the linseed oil hydroperoxides converts organic sulfur into an inorganic form which is extracted by aqueous base. Thereby, the treatment selectively removes organic sulfur with minimum loss of BTU.

## CONCLUSIONS AND RECOMMENDATIONS

### Conclusions

- Pretreating IBC-108 coal with alkali removes 13% to 23% of the sulfur under the conditions of this investigation.. Higher temperatures and higher base concentrations remove more sulfur. Treatments with 5%  $\text{NH}_4\text{OH}$  at 100 °C in the presence of  $\text{O}_2$  or with 5%  $\text{NaOH}$  at 25 °C remove more sulfur (21-23%) than any other treatment with alkali alone.
- More sulfur is removed from the pretreated coal by linseed oil treatment followed by base extraction. The best result (about 40% sulfur removal) is obtained with the combinations of 5%  $\text{NaOH}$ -OIL-5%  $\text{Na}_2\text{CO}_3$  (B-O-C), 1%  $\text{NH}_4\text{OH}$ -OIL-5%  $\text{Na}_2\text{CO}_3$ (Nl-O-C), and 5%  $\text{NH}_4\text{OH}$  (100°C)-OIL- $\text{Na}_2\text{CO}_3$  (N-O-C).
- For the pretreatment,  $\text{NaOH}$  is better than  $\text{NH}_4\text{OH}$  in most experiments and for the final extraction,  $\text{Na}_2\text{CO}_3$  is better than  $\text{NaOH}$ .
- The base-oil-base treatment selectively removes organic sulfur with minimum loss of BTU.

### Recommendations

- Continue with the experimental plan of the proposal using either  $\text{NaOH}$  at 25 °C or  $\text{NH}_4\text{OH}$  at 100 °C for pretreatment and  $\text{Na}_2\text{CO}_3$  at 80 °C for oil extraction.

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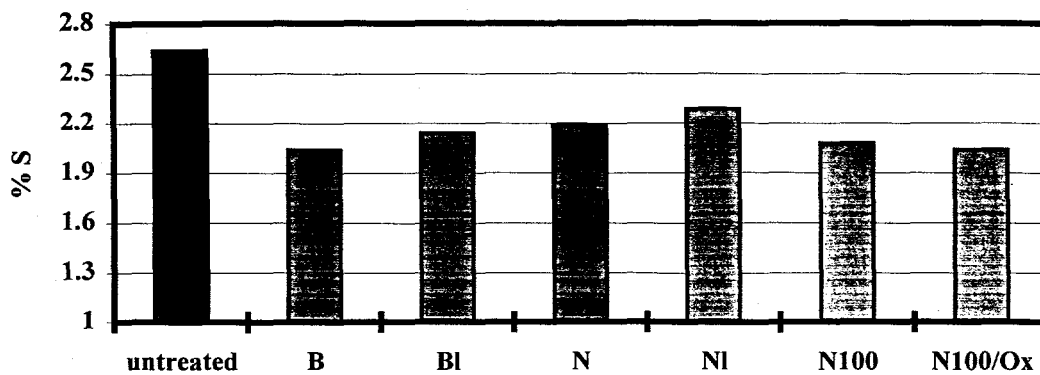
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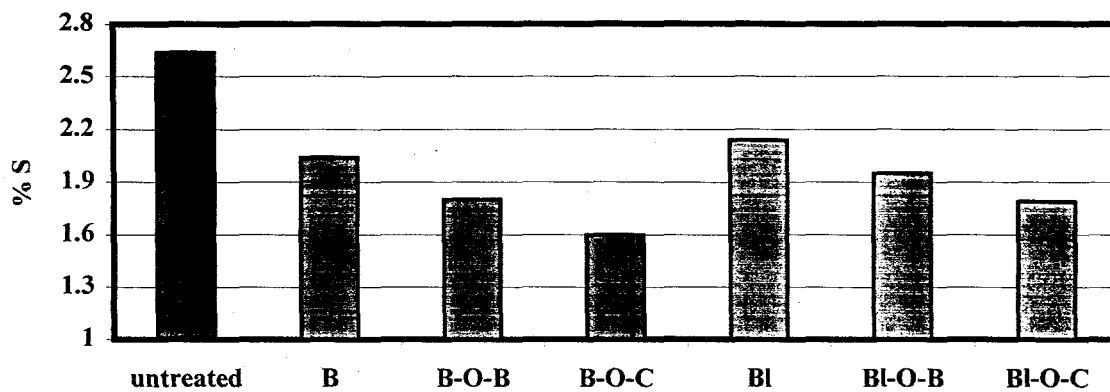
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**Fig 1: % Sulfur Remaining After Treatment with Alkali Under Various Conditions**



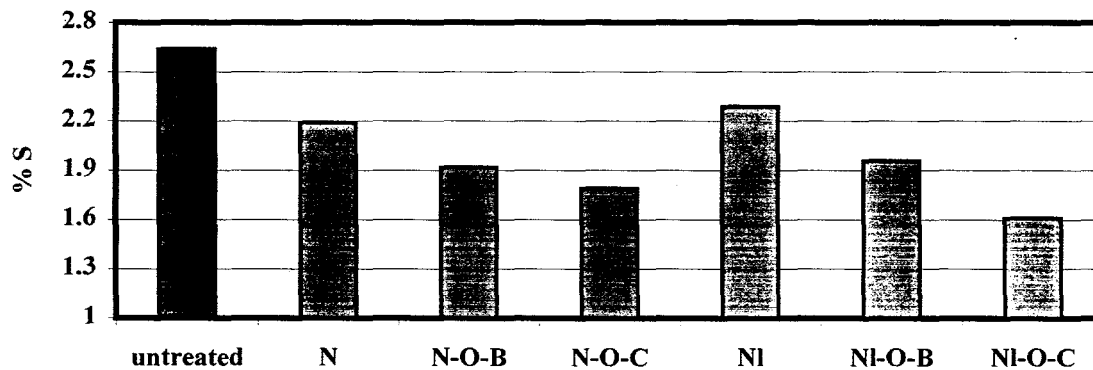
Please see Table 2 footnotes for definitions of B, B1, N, N1, N100, and N100/Ox

**Fig 2: % Sulfur Remaining After Treatment with 5% NaOH and Linseed Oil**



Please see Table 2 footnotes for definitions of B, B1, O, and C.

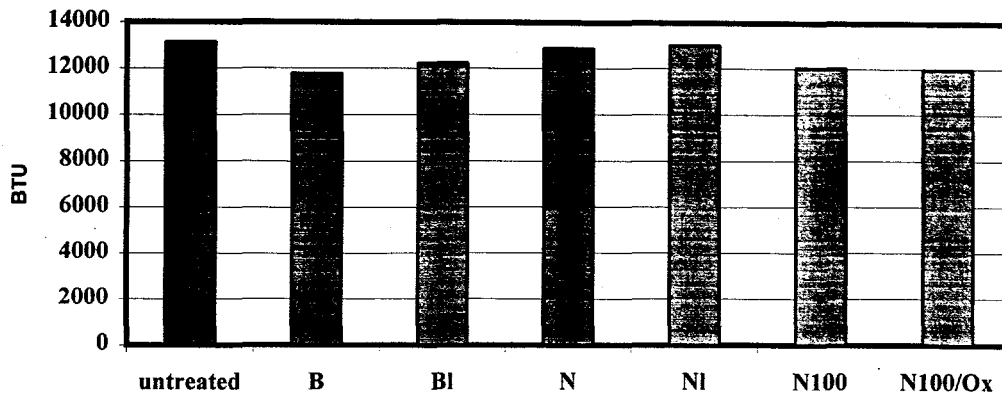
**Fig 3: % Sulfur Remaining After Treatment with NH<sub>4</sub>OH and Linseed Oil**



Please see Table 2 footnotes for definitions of B, N, N1, and O, and C.

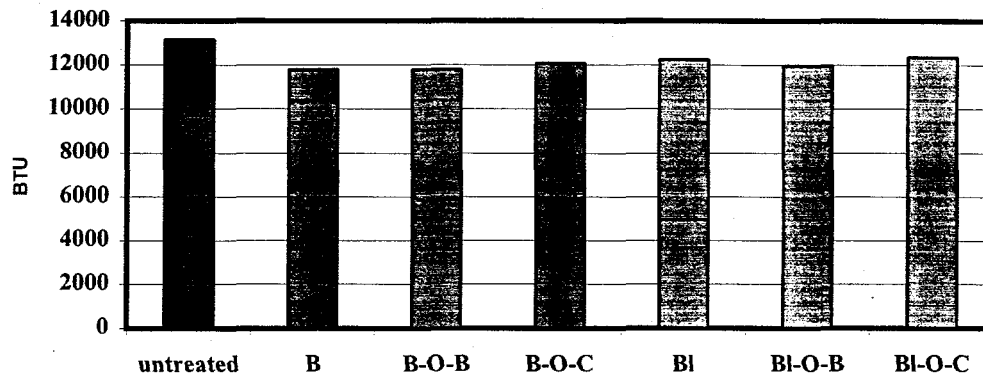


Fig 4: BTU Changes After Treatment with Alkali Under Various Conditions



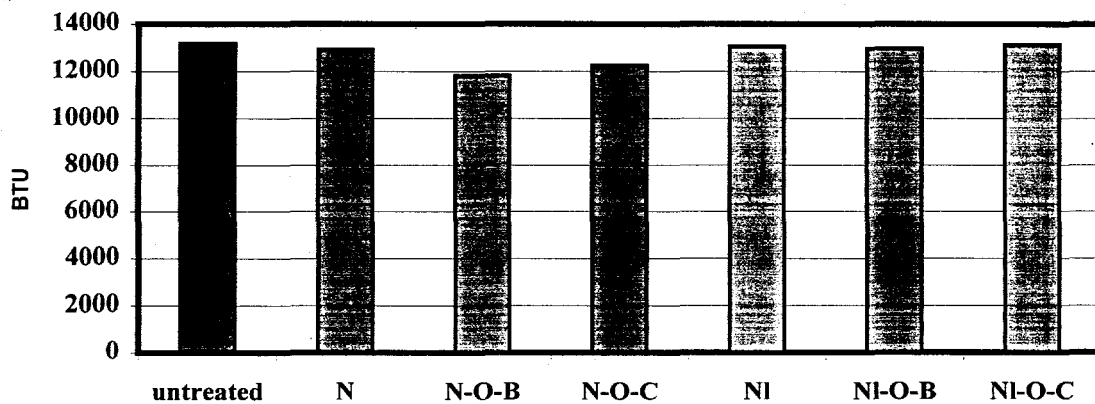
Please see Table 2 footnotes for definitions of B, B1, N, N1, N100, and N100/Ox

Fig 5: BTU Changes After Treatment with 5% NaOH and Linseed Oil



Please see Table 2 footnotes for definitions of B, B1, O, and C.

Fig 6: BTU Changes After Treatment with  $\text{NH}_4\text{OH}$  and Linseed Oil



Please see Table 2 footnotes for definitions of B, N, N1, O, and C.

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

Project Title: **DESULFURIZATION OF ILLINOIS COALS WITH  
HYDROPEROXIDES OF VEGETABLE OILS & ALKALI**

DOE Cooperative Agreement Number: DE-FC22-92PC92521 (Year 4)  
ICCI Project Number: 95-1/1.1D-2P  
Principal Investigator: Gerard V. Smith, Southern Illinois  
University at Carbondale  
Other Investigators: Ricky D. Gaston, Southern Illinois  
University at Carbondale  
Ruozhi Song, Southern Illinois  
University at Carbondale  
Jianjun Cheng, Southern Illinois  
University at Carbondale  
Feng Shi, Southern Illinois  
University at Carbondale  
Project Manager: Ken Ho, ICCI

COMMENTS

Expenditures are proceeding approximately as projected. No unusual problems have occurred.

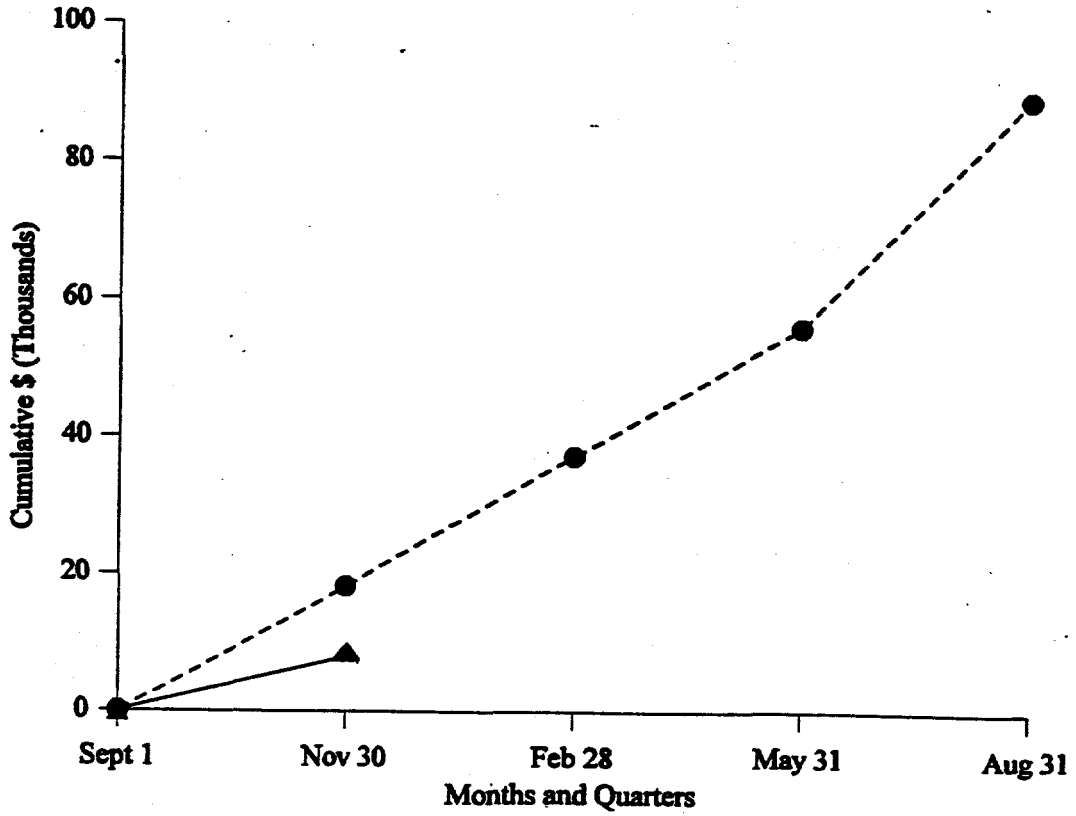
**PROJECTED AND ESTIMATED EXPENDITURES BY QUARTER**

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	12,717	1,730	750	0	0	1,426	1,662	18,285
	Estimated	6,000	641	568	0	0	384	759	8,352
Sept. 1, 1995 to Feb. 28, 1996	Projected	25,434	3,461	1,500	600	0	2,852	3,385	37,232
	Estimated								
Sept. 1, 1995 to May 31, 1996	Projected	38,151	5,191	2,250	1,200	0	4,279	5,107	56,178
	Estimated								
Sept. 1, 1995 to Aug. 31, 1996	Projected	61,545	8,885	3,000	2,000	0	5,705	8,114	89,249
	Estimated								

\*Cumulative by Quarter

### CUMULATIVE COSTS BY QUARTER

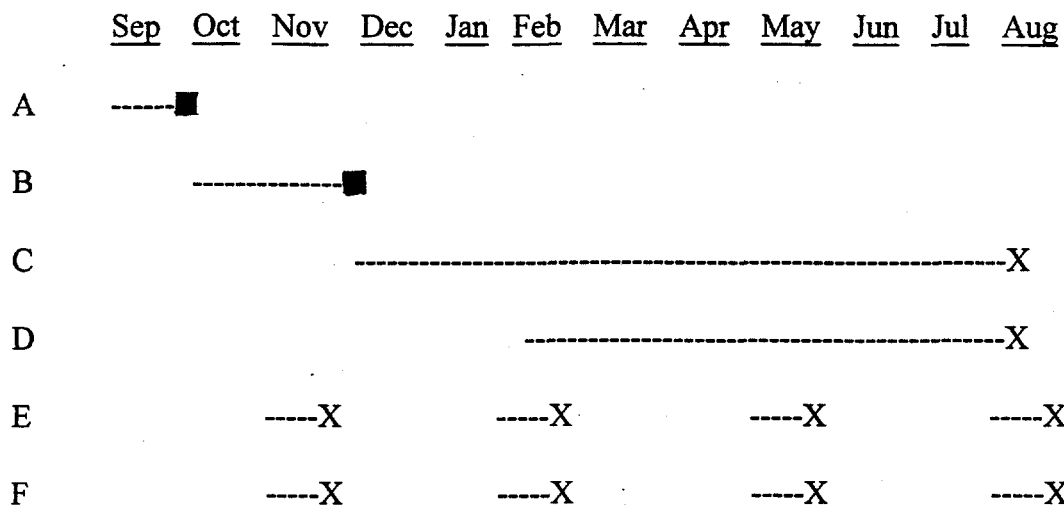
Desulfurization of Illinois Coals with Hydroperoxides of Vegetable Oils & Alkali



● = Projected Expenditures - - - - -  
▲ = Actual Expenditures \_\_\_\_\_

Total Illinois Clean Coal Institute Award: \$89,249

### SCHEDULE OF PROJECT MILESTONES




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Begin  
Sept. 1  
1995

#### Hypothetical Milestones:

- A: Research personnel employed
- B: Task 1, selection of base for pretreatment and extraction
- C: Task 2, determination of base and linseed oil ability to remove organic sulfur from IBC-108 coal
- D: Task 3, determination of mass balance, volatiles, and BTU
- E: Project Technical Reports
- F: Project Management Reports

