

DOE/PC/90052--T2
SEP 13 1990

DOE/PC/90052--T2

DE92 001612

STEAM PRETREATMENT FOR COAL LIQUEFACTION

First Quarterly Report

For the Period

26 September 1990 to 31 December 1990

Robert A. Graff

Valeria Balogh-Nair

The City College of CUNY
Office of Research Administration
New York, NY 10031

Work Performed Under USDOE Contract No. DE-AC22-90PC90052

William E. McKinstry, Program Manager

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

Yoz

ACKNOWLEDGEMENTS

Substantial contributions to the work described here were made by Graduate Research Assistants Olga E. Ivanenko and Claude Brathwaite and Technicians William Hall, Ivan Oritz, Zhen Rong Xu, and Russell Smith.

ABSTRACT

Steam pretreatment is the reaction of coal with steam at temperatures well below those usually used for solubilization. The objective of the proposed work is to test the application of steam pretreatment to coal liquefaction. This quarter, a 300 ml stirred autoclave for liquefaction tests was specified and ordered, procedures for extraction tests were reestablished, and the synthesis of four model compounds was completed. Two of these compounds remain to be purified.

TABLE OF CONTENTS

INTRODUCTION	1
CONSTRUCTION OF LIQUEFACTION TEST APPARATUS	2
PRETREATMENT STUDIES	2
MODEL COMPOUND STUDIES	2
WORK PLANNED FOR NEXT QUARTER	3

INTRODUCTION

This is the first quarterly report of a two year program on the application of steam pretreatment to the direct liquefaction of coal. Steam pretreatment is the reaction of coal with steam at temperatures well below those usually used for solubilization.

Steam pretreatment has been shown to be effective in coal pyrolysis. For steam pyrolysis, it has more than doubled the liquid yield, reduced the molecular weight of pyrolysis liquid by 31%, and increased yields in mild extraction. Studies of pretreated Illinois No. 6 coal indicate that steam reacts with the ether linkages in coal, replacing them with hydroxyl groups. The result is a partially depolymerized coal. The oxygen content of this pretreated coal is 27% that of the feed.

These results suggest that steam pretreatment prior to solubilization will be beneficial to the coal liquefaction process. It is the objective of this work to test this application. Direct liquefaction of steam pretreated coals will be carried out in a stirred autoclave and the results compared with those from the liquefaction of raw coal.

It is also an objective of this work to develop an improved understanding of the chemistry of steam pretreatment. For this purpose, model compounds will be reacted with steam under the same conditions as used for coal pretreatment and their products analyzed to determine reaction pathways.

CONSTRUCTION OF LIQUEFACTION TEST APPARATUS

Autoclave designs offered by the leading manufacturers were examined. A 300 ml stirred autoclave, model DD0300SS03AF16D, manufactured by Autoclave Engineers, Erie, PA, was selected and a purchase order placed with the company.

The pressure vessel will be constructed of Hastelloy C and have a low torque closure with a reusable metallic double delta seal. It will be equipped with a packless magnetic stirrer drive. As a special modification from the standard design, the autoclave will be provided with an external cooling coil of 1/4-inch copper tubing and a custom designed furnace to accommodate this coil. Delivery is expected in January.

PRETREATMENT STUDIES

Several preliminary extraction tests were carried out using raw Illinois No. 6 coal in order to establish experimental procedures.

MODEL COMPOUND STUDIES

The first two model compounds, α - and β -benzyl naphthyl ethers (1 and 2), were synthesized in good yields using a modification of the procedure of Maslak and Guthrie [J. Am. Chem. Soc., 108, 2637 (1986)].

These compounds were prepared by stirring benzyl chloride (0.879 g, 6.9 mmol) with the appropriate naphthol (1.0 g, 6.9 mmol) and K_2CO_3 (2.0 g) in 30 ml dry DMF at 25°C under a nitrogen atmosphere for 36 hours. The reaction mixture was then poured into a H_2O/CH_2Cl_2 mixture (200 ml each). The organic layer was separated and the solvent removed by vacuum. The residue was recrystallized twice from ethanol and checked for purity using thin layer chromatography on hptlc plates. The yields for 1 and 2 were 83% and 67% respectively. The products gave melting points and 1H nmr spectra essentially identical with those reported in the literature. Further purification gave about one gram of each sample with greater than 99% purity as determined by HPLC.

The second pair of compounds, α - and β -naphthylmethyl phenyl ethers, was synthesized in good yields following the same procedure. These compounds are now being purified.

WORK PLANNED FOR NEXT QUARTER

A selection of coals will be ordered from the Penn State Sample Bank. Procedures for pretreatment will be reestablished and a new procedure suitable for model compounds will be developed. Purification of model compounds will be continued.

END

**DATE
FILMED**

12/03/91

