TECHNICAL REPORT
December 1, 1994 through February 28, 1995

Project Title: NOVEL CARBONS FROM ILLINOIS COAL FOR
NATURAL GAS STORAGE

DOE Cooperative Agreement Number: DE-FC22-92PC92521 (Year 3)
ICCI Project Number: 94-1/4.2B-6M
Principal Investigator: Massoud Rostam-Abadi, ISGS
Other Investigators: Jian Sun, Anthony A. Lizzio, ISGS
Mohammad Fatemi, Amoco Research Center
Daniel Banerjee

ABSTRACT

The goal of this project is to develop a technology for producing microengineered adsorbent carbons from Illinois coal and to evaluate the potential application of these novel materials for storing natural gas for use in emerging low pressure, natural gas vehicles (NGV). The focus of the project is to design and engineer adsorbents that meet or exceed the performance and cost targets established for low-pressure natural gas storage materials. Potentially, about two million tons of adsorbent could be consumed in natural gas vehicles by year 2000. If successful, the results obtained in this project could lead to the use of Illinois coal in a growing and profitable market that could exceed 6 million tons per year.

During this reporting period, a series of experiments were made to evaluate the effect of coal pre-oxidation, coal pyrolysis, and char activation on the surface area development and methane adsorption capacity of activated carbons/chars made from IBC-102. The optimum production conditions were determined to be: coal oxidation in air at 225°C, oxicoal (oxidized coal); devolatilization in nitrogen at 400°C; and char gasification in 50% steam in nitrogen at 850°C. Nitrogen BET surface areas of the carbon products ranged from 800-1100 m²/g. Methane adsorption capacity of several Illinois coal derived chars and a 883 m²/g commercial activated carbon were measured using a pressurized thermogravimetric analyzer at pressures up to 500 psig. Methane adsorption capacity (g/g) of the chars were comparable to that of the commercial activated carbon manufactured by Calgon Carbon. It was determined that the pre-oxidation is a key processing step for producing activated carbon with high surface area and high methane adsorption capacity. The results obtained to date are encouraging and warrant further research and development in producing tailored activated char from Illinois coal for natural gas storage.

"U.S. DOE Patent Clearance is NOT required prior to the publication of this document"
EXECUTIVE SUMMARY

Background

An adsorbed natural gas (ANG) storage system is being considered as an on-board storage technology for natural gas vehicles (NGV) because it is less expensive, lighter, occupies less space, costs less to refuel, and has much greater storage density at low pressure (< 35 atm) than that of compressed natural gas (CNG) storage (Golovoy, 1983; Wegrzyn et al., 1992; Nelson, 1993). Carbon-based adsorbents have been found to have the most favorable gas storage density compared to other adsorbents, e.g., zeolites (Golovoy, 1983). A key issue in commercial development of an ANG storage technology is the availability of low cost carbon adsorbents (< $2/lb) with high capacity for methane storage (Nelson, 1993). In addition, advanced cost-effective carbon adsorbents with sieving properties are needed to preferentially remove impurities (CO, CO₂, N₂, C3-C4 hydrocarbons) from natural gas. Illinois coal is a low-cost starting material that has the potential of being an ideal precursor for synthesis of carbon-based adsorbents with desirable properties for ANG technology.

NGV are presently being developed by numerous organizations throughout the world in hopes of capitalizing upon their inherent advantages over gasoline and other alternative fuels. To date, NGV have been implemented mainly in the form of high-pressure compressed natural gas retrofit. However, with the development of adsorbed natural gas storage, advancements are now possible in dedicated vehicle technology as opposed to current compressed natural gas technology, which limits market opportunities available to NGV due mainly to safety concerns. Adsorbed NGV (ANGV) operate at 15-30% of the pressure of compressed NGV. In the U.S., the utilization of ANGV in fleet applications will likely be their initial entry point into the marketplace. Fleet vehicles (taxis and buses) usually accommodate high mileage and often refuel at a central location. This allows usage of dedicated natural gas refueling facilities and maximizes the cost savings between gasoline and natural gas. The U.S. DOE recently compiled statistics which showed the potential market for fleet NGV to be tremendous (Biederman et al., 1989). The current number of total fleet automobiles and private and commercial light trucks is 48 million. This is expected to rise to 60 million vehicles by the year 2000. These statistics reflect a growing market potential for ANGV of up to 12 million units. This study also showed that Chicago, Illinois would be the ideal market for fleet vehicles, and deemed the city the prime U.S. location to begin marketing ANGV.

The potential benefit of the development of adsorbed natural gas technology to the Illinois coal industry is significant just taking into account the expected increase in fleet vehicles. If by the year 2000, twelve million fleet ANGV are added and were to carry an average of 300 pounds carbon adsorbent for low pressure natural gas storage, it would necessitate a demand for 3.6 billion pounds of low-cost carbon. If this carbon were to be made of coal, the demand for a suitable coal would be 12 billion pounds assuming that 30% of the coal is utilized in the final product. Thus, 6 million tons of additional coal would be needed. These calculations do not account
for additional demand due to ANGV entering the main marketplace, where over 250 million units are now in operation. If ANGV find entry into this market, it could mean an even greater demand for coal. The results of research directed towards the development of suitable adsorbent carbons from Illinois coal for natural gas storage should be of great interest to the Illinois coal industry. Based on past history of the auto industry, it is reasonable to assume that if a suitable feedstock for a NGV carbon adsorbent is identified, cost conscious auto makers will rush to adopt use of this material for their adsorbed natural gas vehicles.

Goals and Objectives

The overall goal of this project is to develop a technology for producing microengineered adsorbent carbons from Illinois coal and evaluate the potential application of these novel materials for use in the emerging low pressure ANGV. The focus of the project is to design and engineer adsorbents from Illinois coal that meet or exceed the performance and cost targets established for low-pressure natural gas storage materials.

The project consists of six tasks. In Task 1, chars are prepared from IBC-106 coal in a fixed-bed or a fluidized bed under a wide range of heat treatments and soak times. The pore structure of the chars is tailored for optimum methane storage capacity (about 10-12 Å) via activation in CO₂ or H₂O. Carbon adsorbents with molecular sieve properties will be made by further modifying the pore structure by carbon deposition with CH₄ or by a novel oxygen deposition method. In Task 2, the physical and chemical properties of the products are evaluated to gain additional insight into the fundamentals of preparation and properties of CMS. In Task 3, the methane adsorption capacity (energy storage density) of products and the heat of adsorption will be measured at pressures ranging from 1 to 35 atm with a pressurized thermogravimetric analyzer and by differential scanning calorimetry at the ISGS. Experiments will also be performed at Amoco Research Center (Naperville, IL) with a high pressure volumetric apparatus, designed and constructed for screening adsorbents for ANG, to compare the engineering properties and storage capacity of the adsorbents made from Illinois coal with the commercially available counterparts. In Task 4, the molecular sieve properties of the products are determined by measuring the kinetics of adsorption of O₂, N₂, CO₂, CH₄ and C₂H₄ on chars at 25°C. The ability of the chars to separate impurities from natural gas is tested in an adsorption column/gas chromatography/mass spectrometry system. In Task 5, a process flowsheet for production of adsorbent carbons from Illinois coal suitable for ANGV, is developed, and a technical/economic study is initiated. In Task 6, technical and management quarterly reports are prepared and submitted to the ICCI.
Results and Discussion

Since one of the key issues in commercial development of the ANG storage technology is the availability of low cost adsorbents, the initial focus of this research is to evaluate potential process schemes that would lower the production cost of the adsorbent carbons. Coal pre-oxidation is a necessary and key processing step to manufacture activated carbon/char from Illinois coal.

Twelve activated char samples were made under different production conditions. These experiments were designed to evaluate the effect of coal pre-oxidation temperature and reaction time, pyrolysis temperature, and activation temperature and reaction time on activated char surface area development and methane adsorption capacity.

Depending on the process conditions, activated carbons/chars with surface areas ranging from 691 to 1056 m²/g were made. Air pre-oxidation at 250°C caused excessive burn-off of the outside surface area of coal particles. The optimum production condition for IBC-106 was determined to be: coal oxidation in air at 225°C, oxiccoal (oxidized coal) devolatilization in nitrogen at 400°C, and char gasification in 50% steam in nitrogen at 825 to 850°C.

A pressurized thermogravimetric analyzer was used to measure the methane adsorption capacity (g/g) of selected activated carbon/char at pressures ranging from 0 to 500 psig. The methane adsorption capacities ranged from 0.0474 g/g to 0.0541 g/g. The results for two of the activated chars and a commercial carbon are shown in Figure A. Sample C-n400(1)850(1.5) was made from IBC-106 without pre-oxidation and had a surface area of 897 which is comparable to that of the Calgon BPL (commercial activated carbon). This activated char exhibited a lower methane adsorption capacity than that of the BPL. The activated char prepared from the pre-oxidized coal, C-225(4)400(1)850(2) had a surface area of about 1000 m²/g and a methane adsorption capacity comparable to that of the BPL carbon (0.0505 g/g).

Further analysis of data indicated that there was a correlation between the surface area and the pre-oxidation time up to four hours. It also appeared that there was a correlation between the surface area of the activated chars made from the pre-oxidized coal and the methane adsorption capacity. These results are very encouraging and warrant further research and development in producing tailored activated char from Illinois coal for natural gas storage.

Work is in progress to measure the methane adsorption capacity of samples prepared during this reporting period. Additional research is needed to fully understand the role of coal pre-oxidation in producing carbons with high methane adsorption capacity. In order to improve gas-solid contact during the pre-oxidation step, a one inch fluidized bed reactor will be used. Methane working capacity (volume methane per volume of carbon bed) of activated carbon/char samples are also being measured.
Figure 1. Methane adsorption onto activated carbon
OBJECTIVES

The overall goal of this two-year project is to develop a technology for producing microengineered carbon adsorbents from Illinois coal and to evaluate the potential application of these materials for use in the emerging low pressure natural gas vehicle (NGV). The main objective of the proposed research is to synthesize microengineered adsorbents, from Illinois coal, suitable for use in ANGV which meet the following criteria: a) natural gas storage density of > 150 V/V compared to current 100-120 V/V; b) lower adsorbent cost ($<2/lb). Another important objective is to develop an adsorbent carbon with sieving properties to be used in a guard bed system for controlling the adsorbable contaminants present in natural gas ($C_2-C_4$ hydrocarbons, $N_2$, $CO_2$, odorant, water). The tasks of the project are:

Task 1. Adsorbents are prepared from IBC-106 coal in a fixed-bed or a fluidized-bed reactor under a wide range of heat treatment and activation conditions. The pore structure of the chars is tailored for optimum methane storage capacity (about 10-12 Å) via chemical activation with KOH or activation in CO$_2$/H$_2$O. The pore structure may be further modified by carbon deposition with CH$_4$ to achieve proper molecular sieving properties.

Task 2. Adsorbents are characterized for their physical and chemical properties, including N$_2$-BET and CO$_2$ surface area, porosity and pore size distribution and bulk density, to gain insight into the fundamentals of preparation and the properties of products.

Task 3. Molecular sieve properties of selected products are determined by measuring the kinetics of adsorption of $O_2$, $N_2$, $CO_2$, CH$_4$ and $C_2H_6$ on chars at 25°C. The ability of the chars to separate impurities from natural gas is tested in an adsorption column/mass spectrometry system.

Task 4. Methane adsorption capacity (energy storage density) of products and the heat of adsorption are measured at pressures ranging from 1 to 35 atm. High-pressure methane capacities of products is determined with a high-pressure volumetric adsorption apparatus at Amoco Corporation and results are compared with commercial products tested for ANG.

Task 5. A process flowsheet for production of microengineered adsorbents from Illinois coal is developed. A technical and economic study as well as a market analysis will be initiated.

Task 6. Technical and management quarterly reports are prepared and submitted to ICCI.
INTRODUCTION

Carbon-based adsorbents have been found to have the most favorable gas storage density compared to other adsorbents, e.g., zeolites (Golovoy, 1983). In ANGV applications, the storage volume is often limited (gas tank). Thus for adsorbents with the same capacity per unit mass, the one having the greater packing density in the storage vessel will result in a greater storage capacity. However, adsorbents with lower packing density, but greater capacity per unit mass, may have greater storage capacities. Matranga et al. (1992) performed Monte Carlo calculations to simulate the adsorption of natural gas on carbon. Adsorption isotherms, storage capacity, and isoteric heats of adsorption were determined from model simulations and compared with experimental data. Their model simulations predicted a maximum storage capacity at 35 atm of 244 V/V for monolithic carbon and 169 V/V for pelletized carbon. (The standard terminology for natural gas storage capacity is V/V or volume of stored material at STP per volume of storage container.) Generally, two approaches are taken to optimize storage capacity using carbon adsorbents. An adsorbent with a high mass uptake but low density, such as the Amoco KOH type carbons, can be treated with binder and compacted to improve packing density and thus increase volumetric storage capacity. On the other hand, a high density but low microporosity carbon can be activated in an attempt to increase the microporosity, and thus storage capacity. However, this will be at the expense of decreasing density. For natural gas storage, macroporosity in carbons is undesirable. Amoco type carbons have large macropore volumes which still exist after compaction. This results in reduced volumetric storage capacity. Conventional activation of coal-derived carbons by air, carbon dioxide or steam, also results in macropore formation. Therefore, to minimize macropore formation and increase gas storage capacity, new or modified methods for the preparation of both types of carbons must be found.

EXPERIMENTAL

Coal Preparation

The adsorbent carbons produced in this work were prepared from an Illinois Basin Coal, IBC-106. Twenty pounds of this coal was obtained from the coal bank located at the ISGS. The parent coal was subjected to a step-wise grinding and sieving technique which involved grinding the sample in a rock mill and a Homes mill in order to reduce its particle size from -8 mesh to -20+100 mesh. Size range of -20+100 mesh is considered as that of Granular Activated Carbon. About 20 aliquot of ~5 grams coal sample was prepared by riffling from a 5 pounds portion of the fresh ground coal.

Carbon Production

An oxidation-pyrolysis-gasification reactor system assembled during the first reporting period was used for char production experiments. In a typical run, the sample holder was filled with about 5 gm coal and the sample was placed into the reactor tube, the
air flow was set at 1 liter/min and the furnace was set to 225°C. Under these conditions, the gas temperature above the boat was about 225-250°C. After a predetermined oxidation time, the gas supply was switched from air to nitrogen at 1 liter/min and allowed the nitrogen purge the reactor for at least five minutes. The furnace was set to 400°C to begin the devolatilization step. The reaction temperature reached 425°C and was hold for 30 minutes to one hour (desired devolatilization time). In the gasification step, the nitrogen flow rate was reduced to 0.5 liter/min and the peristaltic pump was turned on with a water flow rate of 22 cc/min (desired water flow rate to produce 50% steam in nitrogen gasification gases). The furnace was set to 800-850°C and hold at this temperature for the desired period of activation time. After the completion of the activation step, the reactor was cooled under a nitrogen purge to the ambient temperature and the sample was stored in a glass bottle.

Carbon Characterization

Single-point BET surface areas of the carbons produced in this work were determined from nitrogen (77 K) adsorption data obtained with a Monosorb flow apparatus (Quantachrome Corp.). A pressurized thermogravimetric analyzer was used to measure methane adsorption capacity of the carbon products at pressures up to 500 psig.

RESULTS AND DISCUSSION

Since one of the key issues in commercial development of the ANG storage technology is the availability of low cost adsorbents, the initial focus of this research is to evaluate potential process schemes that would lower the production cost of the adsorbent carbons.

Activated Carbon/Char Production

Reducing the caking behavior of Illinois coal is a critical factor in producing activated carbon/char with acceptable properties from this resource. Low temperature (200-250°C) pre-oxidation of coal is the most common method to reduce agglomeration tendency of a caking coal. During the first quarterly period, activated chars with surface areas between 700 and 800 m²/g were prepared from IBC-106 using a three step process consisting of pre-oxidation, pyrolysis, and steam activation. The pre-oxidation step was carried out in a sample boat which was located inside a tubular reactor purged with air. In this arrangement a poor gas-solid contact was obtained. Because a key factor during the pre-oxidation step is how well the oxidizing gas (air) is in contact with the coal particles, a new sample boat was designed and fabricated to improve gas-solid contact. The new sample holder consists of a sample basket and a ceramic boat. The sample basket was constructed with two layers of stainless steel cloth (a layer of 200 mesh and a layer of 60 mesh) which is supported by a stainless steel rack placed on the top of the ceramic boat. The screen basket is about 1.25 inch long, 1.5 inch wide and 0.75 inch high.
To compare the effect of pre-oxidation on the surface area of the activated char, a series of experiments were made without the pre-oxidation step. The activated carbons/chars were produced by pyrolyzing coal (20 x 100 mesh) in nitrogen at 400°C, and steam activation in 50% steam at 850°C. Figures 2 and 3 show the coal weight loss, on the dry ash free basis, and carbon surface area, on dry basis, as a function of activation time. There is a strong linear correlation between the weight loss and activation time after one hour gasification time. It appears that during the initial stages of the gasification reaction, the most reactive carbon sites in the char's structure are burning off. The carbon surface area increases with carbon burn off. The maximum surface area is reached after 1.5 to 2.0 hours steam gasification corresponding to 75-80% (daf) carbon burn off, Figure 4. Based on the results of this series of experiments, the degree of char burn-off was used to control the gasification time length in subsequent experiments.

Table 1 lists a number of carbons produced under different production conditions. The carbons are identified as C-oxidation temperature (oxidation time) devolatilization temperature (devolatilization time) activation temperature (activation time). For the runs presented in Table 1, devolatilization at 400°C was chosen instead of that at 500°C. It is possible that at a lower devolatilization temperature a higher concentration of the oxygen functional groups deposited during the pre-oxidation step remain on the coal surface which helps to minimize the caking phenomena. Coal oxidation at 250°C was tested in an attempt to reduce the oxidation reaction time.

Depending on the process conditions, activated carbons/chars with surface areas ranging from 691 to 1056 m²/g were made. Air pre-oxidation at 250°C caused excessive burn-off of the outside surface area of coal particles. The optimum production condition for IBC-106 was determined to be: coal oxidation in air at 225°C, oxicoal (oxidized coal) devolatilization in nitrogen at 400°C, and char gasification in 50% steam in nitrogen at 825 to 850°C.

To evaluate the effect of the coal oxidation on carbon surface area development and methane adsorption capacity a series of experiments were performed at 225°C and at reaction times ranging from one to nine hours. As shown in the table, the product made with three hours oxidation reaction time, (C-225(3)400(1)825(1.5), had the highest surface area, 1056 m²/g.
Figure 2. Coal weight loss vs. activation time

Figure 3. Carbon surface area vs. activation time

Figure 4. Carbon surface area vs. coal weight loss
Table 1. List of properties of activated carbons produced during this reporting period

<table>
<thead>
<tr>
<th>Carbon name</th>
<th>Coal weight loss (daf) wt.%</th>
<th>Carbon surface area (dry), m²/g</th>
<th>Methane adsorption at 500 psig, g/g</th>
</tr>
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<tbody>
<tr>
<td>C-n400(1)850(1.5)</td>
<td>75.8</td>
<td>897</td>
<td>0.0424</td>
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<tr>
<td>C-225(1)500(1)850(1.5)</td>
<td>67.5</td>
<td>691</td>
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<tr>
<td>C-225(1)400(1)850(1.5)</td>
<td>69.1</td>
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<tr>
<td>C-250(1)400(1)850(1.5)</td>
<td>71.6</td>
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<td>C-250(3)400(1)850(1.5)</td>
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<td>C-250(3)400(1)850(2)</td>
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<td>C-225(2)400(1)850(1.5)</td>
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<td>C-225(6)400(1)850(1.5)</td>
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<td>C-225(3)400(1)825(1.5)</td>
<td>76.9</td>
<td>1056</td>
<td>0.0509</td>
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</table>

Reference
BPL (Commercial carbon by Calgon)

![Figure 5. Methane adsorption onto activated carbon](image-url)
Methane Adsorption Capacity

A pressurized thermogravimetric analyzer was used to measure the methane adsorption capacity (g/g) of selected activated carbon/char at pressures ranging from 0 to 500 psig. The results are shown in Figure 5. Sample C-n400(1)850(1.5), made from IBC-106 without pre-oxidation has a surface area of 897 m²/g which is comparable to that of the Calgon BPL (commercial activated carbon), however, its methane adsorption capacity is lower than that of the BPL. The activated char prepared from the pre-oxidized coal, C-225(4)400(1)850(2) had a surface area of about 1000 m²/g and a methane adsorption capacity comparable to that of the BPL carbon (0.0505 g/g).

Figures 6 and 7 show the effect of coal pre-oxidation on surface area development and methane adsorption capacity of the activated char products. There is a correlation between the surface area and the pre-oxidation time up to four hours. It also appears that there is a correlation between the surface area of samples made from the pre-oxidized coal and the methane adsorption capacity. These results are very encouraging and warrant further research and development in producing tailored activated char from Illinois coal for natural gas storage.

CONCLUSIONS AND RECOMMENDATIONS

During this reporting period, the research focused on investigating the effect of coal pre-oxidation temperature and reaction time on activated char surface area development and to seek determine the optimum reaction conditions for to producing an activated carbon with high surface area and high methane adsorption capacity. The optimum process conditions for IBC-106 consists of three steps: coal oxidation in air at 225°C, oxicoal (oxidized coal) devolatilization in nitrogen at 400°C and char gasification in 50% steam in nitrogen at 850°C.

Activated carbons/chars with nitrogen BET surface areas ranging from 800 to 1100 m²/g were produced, depending on process conditions. The methane adsorption capacity of Illinois coal derived chars were comparable to that of a 883 m²/g commercial activated carbon.

Work is in progress to measure the methane adsorption capacity of samples prepared during this reporting period. Additional research is needed to fully understand the role of coal pre-oxidation in producing carbons with high methane adsorption capacity. In order to improve gas-solid contact during the pre-oxidation step, a one inch fluidized bed reactor will be used. Methane working capacity (volume methane per volume of carbon bed) of activated carbon/char samples are also being measured.
Figure 6. Carbon surface area vs time of coal oxidation at 225°C

Figure 7. Methane adsorption onto activated carbon vs time of coal oxidation at 225°C
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REFERENCES


PROJECT MANAGEMENT REPORT
December 1, 1994 through February 28, 1995

Project Title: NOVEL CARBONS FROM ILLINOIS COAL FOR
NATURAL GAS STORAGE

DOE Cooperative Agreement Number: DE-FC22-92PC92521 (Year 3)
ICCI Project Number: 94-1/4.2B-6M
Principal Investigator: Massoud Rostam-Abadi, ISGS
Other Investigators: Jian Sun, Anthony A. Lizzio, ISGS
Mohammad Fatemi, Amoco Research Center
Project Manager: Daniel Banerjee

COMMENTS
### Expenditures - EXHIBIT B

**Cumulative Projected and Estimated Expenditures by Quarter**

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<th>Quarter*</th>
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<th>Fringe Benefits</th>
<th>Materials and Supplies</th>
<th>Travel</th>
<th>Major Equipment</th>
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*Cumulative by Quarter*
COST BY QUARTER - EXHIBIT C

NOVEL CARBONS FROM ILLINOIS COAL FOR NATURAL GAS STORAGE

Cumulative Dollars

Projected Expenditures: Actual Expenditures

Total ICCI Award $49,376
**SCHEDULE OF PROJECT MILESTONES—YEAR 1**

<table>
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<th>Milestones</th>
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<td>A. Preparation of carbon adsorbents (Task 1)</td>
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<td>B. Physical and chemical characterization (Task 2)</td>
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<td>C. Methane working capacity/heat of adsorption (Task 3)</td>
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<tr>
<td>D. Molecular sieve properties (Task 4)</td>
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<td>E. Technical/economic/market studies (Task 5)</td>
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<td>F. Reports (Task 6)</td>
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