



# Energeia

## Interfacial Properties of Coal: A Guide to Catalyst Loading and Dispersion for Coal Conversion

## Creating "Basic" Carbon Surfaces

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Electrokinetic and adsorption studies have shown that the loading, dispersion and activities of coal-conversion catalysts can be enhanced by controlling the pH and the charge at the coal-water interface.

### INTRODUCTION

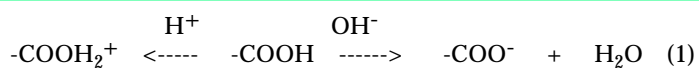
The large coal resources of the United States have been the motivation for research on cost-effective conversion of coal into gaseous and liquid fuels. Advanced technologies for coal gasification and liquefaction can significantly increase coal utilization. However, the fuels produced by these processes must be economically competitive with the price of petroleum. To meet this requirement, a wide range of catalyst-loading techniques have been evaluated in an effort to increase catalyst dispersion and the rates of coal gasification and liquefaction reactions. The common methods of catalyst addition to coal include mixing solid catalyst precursors with coal, exchange of catalyst metal ions with the carboxylic and phenolic protons on coal surface, and the incipient wetness technique, which involves the addition of a sufficient amount of solution containing the catalyst to fill the pore volume of the coal sample. For the same catalytic material, different techniques typically produce different degrees of coal conversion under similar reaction conditions. This suggests disparities in coal-catalyst contact and the attendant differences in catalyst dispersion. Since the majority of coal conversion catalysts are water-soluble, the catalysts are usually loaded from solution. Until recent studies in our laboratory, the surface properties of coal have not been considered during catalyst loading, although the interfacial properties of coal sus-

pensions have been successfully applied to coal beneficiation and to the preparation, dispersion and stability of coal-water slurries. This article examines the effects of coal surface chemistry on the loading of calcium, potassium and molybdenum catalysts onto three ranks of coal. The latter metal is frequently applied to coal liquefaction while calcium and potassium are used for char gasification.

### COAL SURFACE ELECTROCHEMISTRY

Coal contains oxygenated surface functionalities, particularly carboxylic acid and phenolic groups. In aqueous solution, the acidic protons on these groups dissociate, leaving negatively charged sites on the coal surface. The dissociation is enhanced in basic solution, whereas the surface groups are protonated in strongly acidic solution, reducing the negative charge density and the surface can become positively charged in highly acidic solution. Positively charged sites can also develop from proton adsorption by chromene and pyrone-type structures on coal. Charge formation from carboxylic acid group in acidic or basic solution is illustrated in equation (1):

As shown below, the level of catalyst loading and the activity of the catalyst can be enhanced by adjusting the pH and  
(continued, page 2)



One of the largest volume (greater than \$1 billion per year) and oldest applications of carbon is as an adsorbent. Yet, researchers continue to find new means to modify and improve the adsorption properties of this seemingly 'simple' material. Modern work focuses on tailoring the 'surface' chemistry to meet specialized needs. For example, great success has been realized in creating surface acid 'functionalities' that allow carbon to remove only selective chemicals from solutions. Applications include removing undesirable species from flows (e.g. cleaning up waste streams) and recovering and concentrating desired species (e.g. products from a chemical reactor).

Two problems with carbon adsorbents have resisted a solution. First, it has been difficult to create carbons with 'basic' surface chemistry. Yet, carbons with basic surface chemistry could be used, like their acid counterparts, to selectively remove a number of molecules from solutions. Second, it is difficult to create surfaces that resist contamination when exposed to air. Oxygen and water both tend to quickly adsorb on the 'active sites' of the carbon surface, thus reducing the absorbing capacity of the material.

Three researchers at The Pennsylvania State University (PSU), Ljubissa Radovic, Angel Menendez and Jonathan Phillips, recently developed a simple technique to produce a basic and stable (does not adsorb oxygen or water in ambient conditions) carbon surface. Specifically, they found that treating carbon at high temperatures (ca. > 950 °C) in hydrogen creates a surface with a high pH value (PZC>8.5)

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## Interfacial, (continued)

hence, the surface charge density on the coal surface.

### SURFACE CHARGE MEASUREMENTS AND CATALYST ADSORPTION

The surface charge properties of the coals - North Dakota Hagel lignite, Montana Rosebud subbituminous and Illinois No. 6 bituminous coal - were investigated. Slurries for the electrokinetic measurements were prepared by dispersing 300 mg of each coal (-200 mesh) in a liter of deionized water. The zeta potentials (surface charge densities) of the coal particles were determined with Pen Kem model 501 zeta meter after adjusting the pHs of the coal suspensions with HNO<sub>3</sub> or NH<sub>4</sub>OH solution and equilibrating the samples by shaking for 4 hours. Ionic strength was controlled with 10<sup>-3</sup> mol/L AgNO<sub>3</sub>.

The influence of the coal-water interfacial charge on catalyst adsorption was studied by placing 1.0 g of each coal in 50.0 mL of aqueous solution containing 5 x 10<sup>-2</sup> mol/L of calcium, potassium or molybdenum using calcium acetate, potassium carbonate or ammonium heptamolybdate as the sources of the metals. After adjusting the pHs as before, catalyst adsorption was effected by shaking the samples for 24 hours after which they were filtered. The Ca, K or Mo content of each filtrate was measured to determine the extent of catalyst uptake by the coals.

### RESULTS

The zeta potential results in Figure 1 show that, in general, the coals have net negative surface charge densities, as denoted by the negative zeta potentials. However, the charge density increased more rapidly for the lignite than for the subbituminous and the bituminous coals. This trend correlates the oxygen contents of

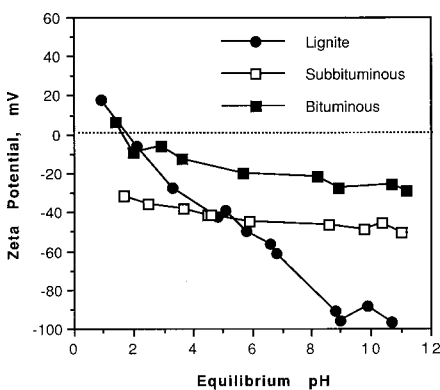


Figure 1. Zeta potentials of the coals.

the coals which were approximately 10, 17 and 22 wt. %, respectively for the bituminous, subbituminous and the lignite and indicates that the coal-water interfacial charge properties are dominated by the dissociation of the protons on the surface oxygen functionalities.

The uptake of calcium by the coals is shown in Figure 2. It is observed that calcium loading onto the lower rank coals (the lignite and the subbitumi-

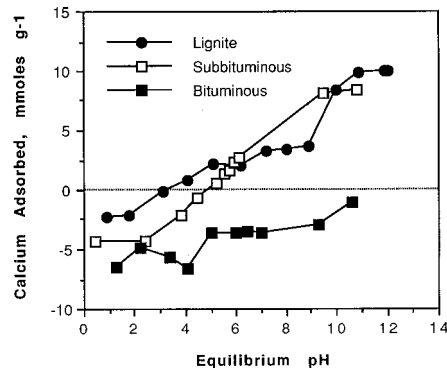


Figure 2. Calcium uptake by the coals.

nous) occurred only above pH ~3 - 5. Similar uptake behavior was obtained for potassium. The adsorption of calcium and potassium ions (Ca<sup>2+</sup> and K<sup>+</sup>) is attributed to electrostatic attraction between the metal ions and the negatively charged coal surface. The negative calcium "adsorption" values indicate extraction of the metal from the coals into solution, resulting in higher concentrations of the metals in solution compared to those of the stock solutions used. Chemical analysis of the coals gave CaO contents of 25, 14 and 7 wt. % for the lignite, the subbituminous and the bituminous coals. Calcium oxide is acid-soluble and the observed increase in calcium concentration of the filtrates is due to the dissolution and extraction of inherent calcium into solution. As shown in Figure 2, calcium adsorption onto the

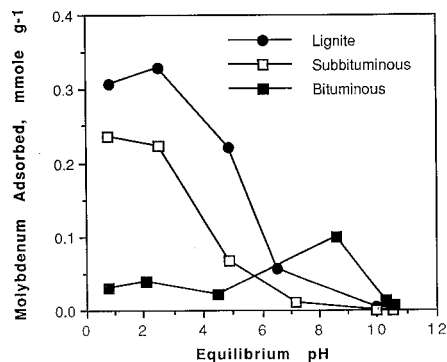


Figure 3. Molybdenum loading onto the coals.

bituminous coal is inhibited at all the pHs investigated, the inhibition being more pronounced in strongly acidic solution than in basic solution.

Figure 3 shows that molybdenum adsorption onto the lower rank coals is promoted in acidic solution but it is suppressed as the solution became strongly basic. Molybdenum loading onto the bituminous coal is generally much lower than on the lower rank coals. Since molybdenum exists as oxyanions (e.g., MoO<sub>4</sub><sup>2-</sup> and Mo<sub>7</sub>O<sub>24</sub><sup>6-</sup>) in solution, the mechanism of adsorption of the molybdenum species onto the negatively charged coal surface is not clear at this time.

After the ion-exchange reactions, it was observed that the filtrates from the acidic slurries were brownish in contrast to the colorless filtrates that were obtained from the basic solutions. Chemical analysis of the filtrates gave high iron concentrations at low pHs. However, iron was practically absent in the filtrates above pH 5 due to the formation of insoluble iron hydroxide. Analysis of the coals also showed that they contained various inorganic materials, including hematite (Fe<sub>2</sub>O<sub>3</sub>). The concentrations of this oxide ranged from about 6 to 24 wt. %, and this supports the extraction of iron and other metals from the coal into solution.

(continued, page 3)

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## Interfacial, (continued)

The inhibition of  $\text{Ca}^{2+}$  or  $\text{K}^{+}$  loading in strongly acidic solution can be explained by the ability of some acids such as nitric acid to dissolve and extract inorganic materials from coal, as was observed in this study.

To minimize the interference from the inorganic constituents, the coals were demineralized by washing in acid solutions. Figure 4 shows the FT-IR spectra of the lignite and its acid-leached samples onto which calcium had been adsorbed at various pHs. A distinct carboxylic acid peak around  $1725\text{ cm}^{-1}$  is observed for the parent coal. However, its intensity decreased progressively as the quantity of calcium exchanged onto the coal increased from 1.3 to 3.2 wt. % Ca. The reduction in peak intensity is ascribed to the exchange of  $\text{Ca}^{2+}$  for the proton on the carboxylic acid group. Similar spectra were obtained for the potassium-loaded samples.

The  $\text{CO}_2$  gasification at  $800^\circ\text{C}$  of the demineralized lignite (DEM 1482) and its samples which were exchanged with calcium at about pH 1, 6 and 10 are shown in Figure 5. Although each coal was contacted with  $5 \times 10^{-2}\text{ mol/L}$  calcium solution, the levels of Ca loading varied significantly: they were 0.3, 4.3 and 7.2 wt. % Ca at approximately pH 1, 6 and 10, respectively. It is observed that the char reactivities increased in the order  $\text{pH } 6 > \text{pH } 10 > \text{pH } 1 > \text{DEM } 1482$ , suggesting that the

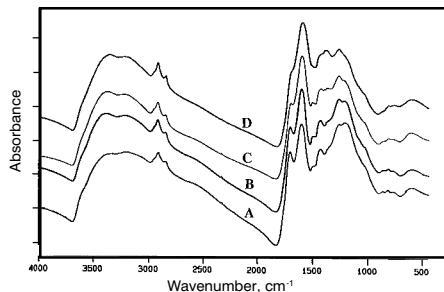


Figure 4. FT-IR spectra of the acid-leached, unloaded lignite (A) and the calcium exchanged samples. Calcium contents, wt. %: (B), 1.3; (C), 2.0; (D) 3.2.

best catalyst dispersion occurred when calcium was loaded around pH 6. This was confirmed by catalyst dispersion measurements using x-ray diffraction analysis. This work has shown that various levels of catalyst loading, dispersion and activity can be attained by controlling the pH and hence, the surface charge

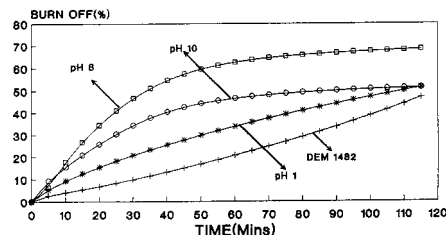


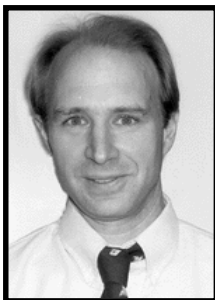
Figure 5.  $\text{CO}_2$  gasification of the lignite char at  $800^\circ\text{C}$ . Ca loadings were 0.3, 4.3 and 7.2 wt. % at pH 1, 6 and 10, respectively. DEM 1482, no added catalyst.

properties of coal during catalyst loading from solution.

Details of this article are provided in *Energy & Fuels* 6, 779, 1992 and 8, 937, 1994 and in *Fuel Sci. Tech. Intl.* 11, 327, 1993.

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## James C. Hower Wins Gordon H. Wood Award



In recognition of his work in Appalachian coal geology, particularly in the areas of coal petrography and geochemistry, and his contributions to colleagues, industry, academia, and students, Jim Hower is this year's recipient of the Gordon H. Wood, Jr., Memorial Award. Founded in 1989, the award was established to recognize outstanding contributions in the field of coal geology and is awarded by the Energy Minerals Division of the Eastern Section of the American Association of Petroleum Geologists.

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## Carbon Surfaces, (continued)

which remains virtually unchanged even after exposure to ambient conditions for months. Previous attempts to generate basic surfaces focused on high temperature treatments in inert gas (e.g. nitrogen). The PSU workers demonstrated that high temperature treatments in inert gases do create 'basic' surfaces as well, but basic surfaces which are very unstable and 'acidify' virtually instantly upon exposure to air.

For many years it has been known that hydrogen treatments create carbon surfaces which have somewhat modified adsorption properties; however, no thorough study was ever conducted. The PSU workers set out to systematically investigate the impact of hydrogen treatments. The only intention was to find new fundamental information about carbon chemistry. Indeed, they discovered a number of new fundamental aspects of carbon chemistry, such as the fact that high temperature hydrogen treatment not only removes adsorbed surface oxygen, but removes highly active 'dangling' carbon atoms as well. The discovery that hydrogen treatments produce a useful new carbon adsorbent was entirely serendipitous.

Five analytical techniques were employed to probe the surface chemistry of the various samples. Microcalorimetric studies, designed to determine both the adsorption capacity of the carbon as well as the strength of adsorption, revealed that high temperature hydrogen-treated samples adsorbed 50 times less oxygen than nitrogen-treated samples. Measures of surface acidity, conducted using two electrochemical techniques ('Point of Zero Charge' (PZC) and 'Isoelectric Point' (IEP)) revealed that the surface of hydrogen-treated samples is basic and that it remains so even after months of air exposure. Adsorption studies revealed that the surfaces of hydrogen-treated carbon is hydrophobic and can successfully remove some chemicals from solution. It was also found that the pore structure and surface area of carbons treated at high temperatures in hydrogen is little different from that of samples treated at a nitrogen treated 'control.' This confirms that changes in adsorption properties relate to the 'molecular' structure of the active sites, and not to the pore configuration.

Enough data was collected to strongly support (not prove) a simple hypothesis regarding the unusual behavior of hydrogen-treated surfaces. To wit: High temperature hydrogen treatments

remove both oxygen groups (as CO and CO<sub>2</sub>), and the 'dangling carbons' which are created during the devolatilization of the carbon oxides. In contrast, high-temperature treatment in nitrogen (or other inert gases) removes oxygen groups, but does not remove the 'dangling carbons' which are created during that process. Thus, only the surfaces created in nitrogen or other inert gases have active sites ('dangling carbons') which readily adsorb oxygen upon exposure to air. The adsorbed oxygen creates acid sites on the surface. Therefore: nitrogen-treated surfaces are not stable, as they rapidly adsorb oxygen and re-acidify upon exposure to air. Hydrogen-treated surfaces cannot adsorb oxygen at room temperature as there are no dangling carbons. 'Active sites' do exist on hydrogen-treated surfaces, but these are only capable of adsorbing oxygen at elevated temperatures. Not surprisingly, significant oxygen adsorption was observed on the hydrogen-treated material at 100 °C. Indeed, it has long been known that active sites can exist on carbon surfaces that only adsorb oxygen at elevated temperatures.

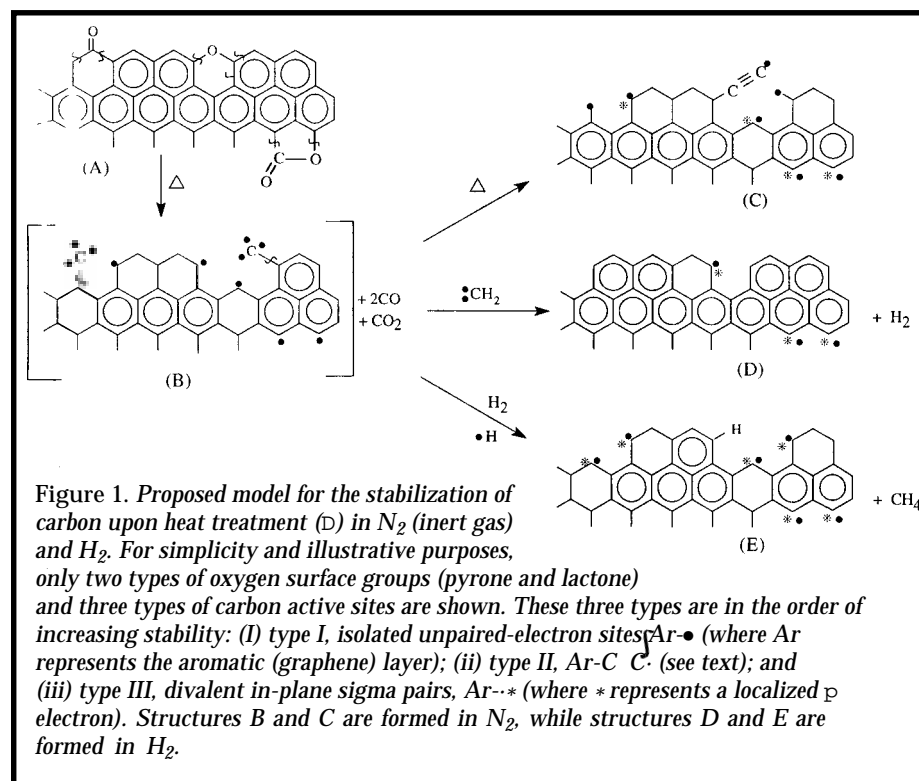
A pictorial representation of the postulated carbon removal process and consequent 'dangling carbon' creation is shown in Figure 1. It shows that the heating process removes surface oxygen in the form of CO (only pyrone and lactone shown) leaving highly unsaturated carbon atoms behind. Thus, a surface like B or C in the figure is

expected to exist after the nitrogen treatment. Clearly, on these surfaces there are carbon atoms that will rapidly adsorb oxygen upon air exposure. The hydrogen treatment removes the dangling carbons (as methane), leaving a surface such as that shown in D or E. The unsaturated sites on this surface, containing, for example, divalent in-plane sigma pairs, will not be as reactive as the 'dangling' carbons found on the nitrogen-treated surfaces.

Several findings suggest that the hydrogen-treated carbons have commercial potential. One clear advantage over other carbons with active sites is that high temperature hydrogen-treated carbon can be shipped and stored without special handling. Another potential benefit is that adsorption can be 'turned on' simply by raising the temperature slightly above ambient and 'turned off' simply by lowering the temperature to ambient. Early adsorption studies are encouraging. Indeed, water isotherms indicate these carbons are significantly more hydrophobic than their nitrogen-treated counterparts. They also were found to adsorb at ambient far more phenol (2x) from solution than the high temperature nitrogen-treated variety.

An additional discovery made by the PSU research group clearly makes the stable, basic surfaces even more attractive economically. These research-

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## COMMENTARY

### COAL—THE MOST IMPORTANT ENERGY SOURCE IN CHINA

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#### SITUATION AND PROBLEMS

In 1994, 1,240 million tons of coal were produced in China, which accounted for 75 percent of the total Chinese energy production. More than 80 percent of consumed coal was burned directly in power plants, industrial furnaces and boilers, as well as, used in household cooking and heating. The Chinese government has placed increasing emphasis on coal conversion and clean-coal utilization since the early 1980's. It has sponsored many important coal related R & D projects, and has taken measures to reduce the emissions caused from coal combustion.

#### COAL COMBUSTION

In some developed countries, most of the coal produced is burned in power plants for electricity. In these cases, it is easier to control the coal's emissions than in China, where only 28 percent of coal produced is burned in power plants, whereas 43 percent is burned in large numbers of small-scale industrial furnaces and boilers, and a little more than 20 percent is burned in households for cooking and heating. The diversity of applications makes environmental protection difficult.

In the larger cities, several strategies have been taken to reduce pollution from coal combustion. 1) Increasing town gas supplies to gradually eliminate the old coal stoves. The town gas sources can be liquified petroleum gas (LPG), coke oven gas and other coal derived gases. 2) Developing co-generation power plants and popularizing hot-water pipeline networks to replace the small boilers for heating systems. Owing to their higher efficiency and lower emissions, these means significantly improve the environment in urban areas.

Particulate collectors have been equipped in most power plants. The flu gas desulfurization (FGD) technology has also been introduced gradually, mainly with imported technologies. For instance, power plants in Chongqing, Taiyuan and Huangdao have been equipped with FGD systems using Japanese technologies. However, the by-product of the Limestone-Gypsum method does not seem to have a market. To stimulate the incentive for industry to equip FGD systems, the by-product of FGD can be sold as a higher-value commodity.

The circulated fluidized bed combustor (CFBC) has been extensively developed in China to improve the environment and efficiency of small and medium scale boilers. The 10 t/h, 35 t/h and 75 t/h CFBCs are commercially available now. A demonstration project for 220t/h CFBC is being carried out at the Institute of Engineering Thermo-physics and Tsinghua University.

The use of coal briquets can increase heating efficiency 10-20 percent and greatly reduce pollution, especially those briquets with sulfur retention agents, which can potentially reduce half of the SO<sub>2</sub> emissions produced from power plants. It is a realistic approach of reducing pollution to increase the production capacity of coal briquets and encourage their use in industrial and residential areas. Research on reducing NO<sub>x</sub> emissions is being carried out in research institutes and universities. New types of de-NO<sub>x</sub> burners and boilers have been developed and some of them have been put into application.

#### COAL DERIVED OIL

To increase the production of oil and gas, the inland and offshore exploration of new oil and gas fields has become quite active. Some newly discovered oil and gas fields are prospective candidates for resource use. However, a big gap will still exist in the next couple of decades. As a supplementary source of petroleum,

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## Carbon Surfaces, (continued)

ers discovered that hydrogen creates stable, basic surfaces at 450 °C (rather than 950 °C) in the presence of a noble metal. That is, simply by mixing a noble metal in with the carbon during the hydrogen treatment, the necessary treatment temperature can be lowered by about 500 degrees!! The hypothesis used to explain this finding is a simple variation on the 'hydrogen spillover' hypothesis first developed to explain the phenomenon in catalysis. To wit: Noble metals dissociate hydrogen molecules. The hydrogen atoms so created are very mobile and diffuse over surfaces. Moreover, these atoms have chemical properties significantly different from molecular hydrogen. For example, in many cases it has been shown that oxides are reduced at far lower temperatures by hydrogen atoms than by molecular hydrogen.

In mixtures of carbon and noble metal (platinum) the PSU group postulated that hydrogen atoms generated by the noble metal diffuse from the noble metal, both via the gas phase and over the surface, to the carbon surface even at low temperatures (ca. 100 °C). However, they perform significant chemistry only at temperatures above about 400 °C. Evidence suggested that two important processes occur at lower temperatures in the presence of hydrogen atoms than in molecular hydrogen: (I) oxygen removal and (II) dangling carbon gasification. As these are the two processes required to create a basic, stable carbon surface, the presence of noble metal in the mix significantly lowers the necessary processing temperature.

#### CONCLUSION

In sum, the PSU workers found a means to create a unique carbon surface. That is, they showed that a relatively low temperature (ca. 450 °C) treatment of carbon (mixed with noble metal) creates an oxygen free, highly basic, and stable surface. These carbons hold potential commercial value for several reasons. First, they have unique adsorption properties, such as enhanced capacity for phenol adsorption from solution, and temperature switch adsorption. Second, they can be shipped and stored without any concern for loss of properties. Third, they contain an unusual set of active sites, which may have particular chemical applications.

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*Dr. Phillips has been in the Chemical Engineering Department at The Pennsylvania State University since 1982.*

## Commentary, (continued)

coal derived oil projects are being carried out in most major coal research institutes and universities.

Since the early 1980's, The Institute of Coal Chemistry (ICC) has been developing a two-stage process of converting syngas into gasoline. A demonstration plant with a capacity of 2,000 tons per year is under construction. Hydrogenation of coal to produce liquid fuel was carried out in the Central Coal Mining Research Institute (CCMRI) under some international cooperative projects. Most Chinese coals were evaluated for their adaptability of hydrogenation. Research on upgrading of coal oil was also conducted. In addition, two coal pyrolysis demonstration plants were constructed in Inner Mongolia with the purpose of producing gas and oil respectively.

The production and application of coal water mixture (CWM) has been developed in China as well. At present, 6 CWM plants exist in China with a total capacity of 1 Mt. CWM annually. The coal in CWM should be highly deashed so that it may act as fuel oil.

## COAL GASIFICATION

Coal gasification is another important subject in coal conversion. China hopes that coal gasification is able to meet all demand for town gas, industrial fuel gas and synthetic gas for ammonia, liquid fuels, and other chemicals. Those cities which are near large refineries use liquefied petroleum gas as town gas. It needs no pipeline network, but is inconvenient and not really safe enough for the residents. Coke-oven gas is the major pipeline town gas in China. Some large cities, such as Beijing, Shanghai, Taiyuan and Dalian, use coke oven gas as town gas. It needs high quality coking coals and the investment per unit Btu of gas is high. Some new gasifiers were equipped for town gas supply in other large cities. For instance, Lurgi gasifiers were installed in Harbin and Lanzhou. It has been quite satisfactory for town gas supplies. In addition, water gas generators combined with methanation processing and two-stage gas generators have also been used in some small cities for town gas.

Most small and medium scale ammonia plants use coal as a feed stock. However, it is low in energy efficiency. Recently, Lurgi and Texaco gasifiers were introduced to produce synthetic gas for ammonia and methanol. Coal derived liquid fuels and chemicals

will be an important developing field in China. Therefore, it is not strange that the C1 chemistry research is very active in Chinese research institutes and universities.

Integrated gas combined cycle (IGCC) will be another application of coal gas. The Chinese IGCC project is in the initial stage of its development. International collaboration will be sought. The Institute of Coal Chemistry is developing an ash agglomerating gasification technology. A pilot gasifier with throughput of 24 tons per day was constructed recently. Now, a demonstration gasifier with throughput of 100 t/day is under construction. The CCMRI has developed a two-stage gasifier for the town gas supply in mining areas.

## SUMMARY

The Chinese people are lucky to have huge coal reserves as an energy resource. On the other hand, it must be recognized that coal is a black, dirty solid which causes many problems in use. Chinese scientists and engineers are making great efforts to improve the clean and efficient utilization of coal. In this respect, international collaboration is definitely necessary.

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