

## **Carbon Sequestration Research in the Office of Science and Technology at the National Energy Technology Laboratory**

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### **Introduction**

The National Energy Technology Laboratory (NETL) is the nation's newest National Laboratory. It has campuses in Pittsburgh, PA, and Morgantown, WV. It is the premier DOE laboratory for fossil fuel research and has a history of more than 75-years of providing science-based, technological solutions to issues associated with the environmental, supply, and reliability constraints of producing and using fossil resources. Since 1993, researchers in the NETL's Office of Science and Technology (OST) have been performing carbon sequestration research. The OST research program has expanded in recent years as concerns about the impact of rising atmospheric CO<sub>2</sub> levels on climate and global ecosystems intensify. A Carbon Sequestration Science Focus Area has been established within OST to foster the development of the growing research program.

Carbon Sequestration Science is a relatively new field. It is remarkably broad-based, encompassing major parts of chemistry, physics, biological and geological sciences, as well as engineering, computational science, and other disciplines. The OST Carbon Sequestration Science Focus Area divides its effort into six major tasks, consisting of 16 individual projects that include 1) Capture and Separation (5 projects), 2) Geological Sequestration (7 projects), 3) Oceanic Sequestration (1 project), 4) Chemical Sequestration (1 project), 5) Geological Sequestration Modeling (1 project), and 6) Process Modeling and Economic Assessment (1 project). Other major areas of Carbon Sequestration Science, such as sequestration in terrestrial ecosystems and biological sequestration, are not addressed in the OST work plans. A conscious, deliberate decision was made early in the planning stage to omit sequestration in terrestrial ecosystems from our efforts. OST intends to initiate work in biological sequestration in the future. The report, *Carbon Sequestration Research and Development* (1), a road mapping document, was used as a guide to frame the overall approach to the work, and as a source to focus individual research projects on specific goals.

### **Task 1. Capture and Separation**

The Capture and Separation task contains five projects that can be divided among those that use dry scrubbing (3 projects), and electrochemical pumping (1), as well as a project whose goal is to develop NETL's facilities to capture and separate CO<sub>2</sub> (1 project). In addition to conducting research to capture and separate CO<sub>2</sub> from conventional flue gas, NETL is also investigating the

separation and capture of CO<sub>2</sub> from gasifiers. Projects within the Capture and Separation Task include the following:

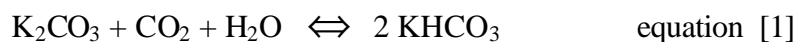
*CO<sub>2</sub> Scrubbing With Regenerable Sorbents*  
*Novel Amine Enriched Absorbents for Capture*  
*Sorbent Development PSA/TSA*  
*Electrochemical Devices*  
*Scoping Exercise: Capture Facility.*

The objective of the project entitled *CO<sub>2</sub> Scrubbing With Regenerable Sorbents* is to identify potential regenerable sorbents that could be used for the capture of CO<sub>2</sub> from a gas stream and to validate a potential dry, regenerable sorbent process that is capable of removing CO<sub>2</sub> from a gaseous stream. The experimental approach taken in this research effort is to utilize a thermogravimetric analyzer (TGA) to track sorbent weight change as the material is exposed to gases under conditions representative of absorption or regeneration. Change in sorbent weight can be linked to the extent of chemical reaction, from which kinetic information can be extracted. Additionally, scaled-up experiments will be performed in a packed-bed reactor to complement the TGA study.

An experimental study was performed to evaluate the potential of alkali- and alkaline-earth metals for use as dry, regenerable sorbents for the capture of CO<sub>2</sub> from a gas stream. Thermodynamic analysis identified ranges of temperature for absorption and regeneration that would be thermodynamically feasible. Potassium carbonate is applicable for CO<sub>2</sub> capture at low absorption temperature (less than 145 °C), while calcium oxide is applicable for much higher absorption temperature (less than 860 °C).

Experiments were conducted in both a TGA reactor and a packed-bed reactor using sorbent fabricated from potassium carbonate supported on a high surface area activated alumina. Sorbent batches with potassium loadings (as potassium carbonate) of 12.2 and 17.1 weight percent were prepared for experimental evaluation. Chemical analyses indicated approximately one-third of the surface area was lost upon impregnation of the metal, but the potassium loading is uniform across the cross-section of the sorbent sample.

A typical weight/time curve for a TGA experiment is shown in Figure 1. This particular experiment used Batch #1 sorbent that was evaluated at an absorption temperature of 80°C. The sample is dried at 150°C in nitrogen for 3-4 hours, followed by humidification, and then CO<sub>2</sub> absorption. Some CO<sub>2</sub> is desorbed (physical sorption) upon removal of CO<sub>2</sub> as an input, and then humidification of the sample is ceased, resulting in additional weight loss as moisture desorbs from the sample as shown in Figure 1. The chemical equation for the capture reaction is shown in equation 1. Considerable time is required for the sample to achieve steady state during each particular phase of the experiment. The experiment depicted in Figure 1 lasted almost four days.



The weight/time data from the TGA can be interpreted in several manners. Preliminary attempts were made to obtain the temporal rate of weight changes during absorption and desorption, but are not reported here. If such information could become available, then the information could be linked to kinetic rate law expressions. An overall capacity of the sorbent proved to be more reproducibly quantifiable. The sorbent capacity is based on the difference in weight, under humidified conditions, of the sorbent after all CO<sub>2</sub>, which was physically sorbed, has been desorbed. In the example of Figure 1, the weight gain (from forming potassium bicarbonate) equaled 26 percent of the theoretical weight gain if all of the potassium carbonate was converted to potassium bicarbonate. Hence the sorbent capacity (i.e., utilization) is reported as 26%.

TGA experiments were typically conducted using a gas composition of 10 mole % CO<sub>2</sub>, 10 mole % H<sub>2</sub>O, with balance N<sub>2</sub>. TGA results indicate CO<sub>2</sub> capture is favored at low absorption temperature (50-60 °C), with sorbent utilization strongly decreasing with higher absorption temperature (80-100 °C). Higher potassium loading on the sorbent did not provide additional benefit for CO<sub>2</sub> capture, as evidenced by lower sorbent utilization for the higher loaded sorbent. The sorbent was thermally regenerated at 150 °C, which is consistent with the predicted temperature based on thermodynamic analysis.

## **Task 2. Geological Sequestration**

The Geological Sequestration task contains seven projects that encompass investigations of CO<sub>2</sub> sequestration in brine fields, in active and depleted oil and gas fields (including natural gas hydrates), and in coal seams. One project, entitled *Sequestration in Brine Fields, Oil and Gas Fields, and Natural Gas Hydrates* attempts to develop an understanding on a macro level of what occurs when large volumes of CO<sub>2</sub> are pumped into a geological formation; while another, entitled *An Investigation of CO<sub>2</sub>/Water/Rock Interactions and Chemistry*, seeks to develop insight into what occurs on a chemical/mineralogical level. This project addresses the aqueous chemistry of CO<sub>2</sub> with brines and rock. With the United States Geological Survey's (USGS) Hydrothermal Laboratory as our partner, NETL is beginning to investigate the uncertainties associated with heterogeneous reactions that may occur with minerals and strata, as well as the uncertainties associated with the complex ionic equilibria and kinetics of CO<sub>2</sub>/water/rock interactions. A second component of this project is concerned with developing a better understanding of low temperature, low pressure formation of carbonate minerals from brine reactions with CO<sub>2</sub> both in the presence, and absence, of rock. Projects within the Geological Sequestration Task include the following:

- Sequestration in Brine Fields, Oil and Gas Fields, and Natural Gas Hydrates*
- An Investigation of Gas/Water/Rock Interactions and Chemistry*
- Chemistry of Carbon Dioxide Sequestration in Coal Seams*
- Sequestration of CO<sub>2</sub> in Coal Seams and Production of Methane Therefrom*
- Comprehensive Monitoring Techniques*
- Scoping Exercise for a Geological Sequestration Simulation Facility (GSSF)*
- Collection of Brines and Surrounding Strata.*

The injection of CO<sub>2</sub> into coal-seams to promote the production of coal-bed methane has recently become of considerable interest to the private sector and the U.S. DOE (1-3). By preferentially absorbing CO<sub>2</sub> onto the coal surface and displacing sorbed methane, this technology may

substantially increase methane production above the level achievable without injection of CO<sub>2</sub> (4). If such increases can be demonstrated, then favorable economics may make coal-seam injection one of the most attractive options for CO<sub>2</sub> sequestration.

The objective of the project entitled *Chemistry of Carbon Dioxide Sequestration in Coal Seams* is to obtain information useful for assessing the technical feasibility of CO<sub>2</sub> sequestration in coal-seams by defining those parameters that affect both the capacity of a coal-seam to adsorb CO<sub>2</sub> and the stability of the formation once formed. The work involves studying the interaction of a number of the Argonne Coal Samples (5,6) with CO<sub>2</sub> under different environmental conditions. The variables of interest are listed in Table 1. The goal is to provide data relating storage capacity and stability to coal and environmental properties, which can then be used as part of the evaluation of whether a candidate seam is appropriate for geologic sequestration. The Argonne coals have been especially prepared to be representative and reproducible from sample to sample. They were mined, ground, thoroughly mixed, and stored under nitrogen.

Gas-phase CO<sub>2</sub> adsorption isotherms were determined using a common manometric technique. The temperature and pressure were maintained at values below the critical temperature and pressure of CO<sub>2</sub> thereby maintaining gas-phase conditions. A reference-cell, contained within a thermostated bath ( $\pm 0.1$  °C), was pressurized to the desired level as indicated on a pressure transducer. The maximum pressure for any given isotherm was limited by the operating temperature and the condensation pressure of CO<sub>2</sub> at that temperature. A sample-cell of known void volume, which was also contained within the same thermostated bath, was pressurized from the reference-cell. Using the change in pressure in the reference-cell and accounting for the gas compressibility, the number of moles of gas transferred from the reference-cell was calculated. Similarly, the gas-phase moles of gas in the sample-cell after the gas transfer were calculated from the post-transfer sample-cell pressure. The missing moles of gas were attributed to adsorption onto (into) the coal.

$$n = (\text{moles transferred from reference}) - (\text{moles in gas-phase in sample-cell}) \quad \text{equation [2]}$$

The reference-cell was then pressurized to a higher pressure and the process repeated. The individual incremental gas adsorption values are summed to generate the adsorption isotherm in a step-wise fashion as shown in Figure 2. The adsorption isotherm for a given temperature is plotted with the total number of millimoles of carbon dioxide adsorbed per gram of coal (y-axis) as a function of the equilibrium sample-cell pressure (x-axis). After completing the experiment at one temperature, the temperature of the thermostated bath was raised, and the process repeated. From the temperature dependence of the adsorption isotherms, the isosteric heat of adsorption was calculated from the modified Clausius-Clapeyron equation:

$$\ln (P_2/P_1) = Q_{\text{isosteric}} (T_2-T_1) / RT_1T_2 \quad \text{equation [3]}$$

In the pressure-temperature region studied, the CO<sub>2</sub> adsorption isotherms appear to be non-Langmuir, Figure 2, in that they fail to approach a limiting value at high pressures, as would be predicted by the Langmuir equation. The incremental amount of CO<sub>2</sub> that can be adsorbed by the coal drops off dramatically at higher pressures. For example, the amount of CO<sub>2</sub> adsorbed

during the application of the first 100 psi of CO<sub>2</sub> pressure (0-100 psia) amounts to 0.6 to 0.9 mmole per gram of coal, depending on the temperature. However, the additional amount of CO<sub>2</sub> adsorbed during the addition of the last 100 psi of CO<sub>2</sub> pressure (600-700 psi) amounts to less than an additional 0.1 mmole per gram. From a practical stand-point, this means that disproportionately higher pumping costs per pound of CO<sub>2</sub> will be incurred at higher sequestration pressures. The effect of increasing temperature is to decrease the equilibrium adsorption capacity of the coal. This is expected because higher temperatures increasingly favor the gas-phase due to the T<sup>a</sup>S entropy term in the free energy expression. This means that otherwise equivalent, but deeper, warmer seams will adsorb less CO<sub>2</sub> than more shallow, cooler ones. The isosteric heat of adsorption was calculated to be 4.85 ± 0.26 kcal per mole of CO<sub>2</sub> adsorbed. This is higher than the heat of vaporization of CO<sub>2</sub> in this temperature range which is only about 1.3 kcal/mol, even at the lowest temperature. It is, however, less than the 12 kcal/mole which has been measured in low-coverage experiments. Thus, it appears that in this case, the binding falls between simple pore-condensation and the higher energy adsorption of the more active sites. On the average, the strength of interaction is about the same as for a typical hydrogen bond (ca. 5 kcal/mol).

### **Task 3. Oceanic Sequestration**

An important issue in determining the fate of CO<sub>2</sub> in the deep ocean is understanding the possible occurrence and impact of the CO<sub>2</sub> clathrate hydrate compound (CO<sub>2</sub> · nH<sub>2</sub>O; 6 < n < 8), hereafter referred to as hydrate, which can form in the ocean as discrete particles or as shells on CO<sub>2</sub> drops at depths below about 500 m (1). Theoretically, pure CO<sub>2</sub> hydrate particles should sink in the ocean (7). This would facilitate sequestration by transporting the CO<sub>2</sub> to even greater depths than used for injection. However, it has been previously demonstrated that hydrate particles will initially float if formed from a two-phase, liquid CO<sub>2</sub>/seawater system, such as would be present in current direct injection scenarios (8). On the other hand, if the CO<sub>2</sub> is first dissolved in the seawater, this single-phase system produces a sinking hydrate upon reaching hydrate-forming conditions (9). If a hydrate shell formed on a CO<sub>2</sub> drop, it would retard the dissolution of the CO<sub>2</sub> into seawater and would therefore frustrate sequestration if the hydrate-encased CO<sub>2</sub> drops rise to shallower depths before dissolving (8). Finally, hydrate formation has recently been shown to rapidly occur in actual experiments in the ocean under the scenario where CO<sub>2</sub> is introduced at depths greater than 3000 m where the CO<sub>2</sub> is more dense than seawater and therefore sinks to the bottom (9). Rather than just forming a protective layer on a CO<sub>2</sub> lake, dynamic hydrate formation resulted in rapid expansion of the CO<sub>2</sub> mass. These examples show that understanding hydrate formation occurrences and processes is therefore critical to successful deployment of strategies to introduce CO<sub>2</sub> into the deep ocean in a manner that leads to long-term sequestration.

NETL is constructing a High-Pressure Water Tunnel Facility (HWTF) that will permit an accurate simulation of the ocean water column encountered by injected CO<sub>2</sub>. In the HWTF a fluid particle, such as a CO<sub>2</sub> drop, is held in an observation section solely by a countercurrent flow of water or seawater. Screens or other restraining devices are not required. Such devices can impact hydrate nucleation and also have unnatural heat transfer characteristics relative to the open ocean. In the HWTF, specialized internal geometries and flow conditioning elements are used to modify the velocity profile in the observation section to provide both axial and radial stability of the fluid particle for extended periods. A generalized schematic of such a water

tunnel device is shown in Figure 3. This device is placed in a flow loop that provides recirculation of water through the system. For a positively buoyant object, the flow of water or seawater enters the top of the water tunnel and passes through a stilling section (not shown in Figure 3). At the end of the stilling section, a flow conditioning element is placed to provide the velocity profile required for radial stabilization of the buoyant object in the observation section immediately below it. The top flow conditioning element shown in Figure 3 represents a bundle of small tubes of different length. Various other configurations are possible. Increasing the length of the tubes in the center results in more head loss in this region and results in flow redistribution with the desired local velocity minimum in the center of the observation section of the water tunnel. The diameter of the observation section increases from top to bottom ( $x_2 > x_1$ ) which provides the downstream axial velocity drop required for axial stabilization. At the exit of the test section, another flow conditioning element may be used. In Figure 3, this lower element depicts another possible tube bundle shape that could be used. A final stilling section is located after the test section (again not shown in Figure 3). To stabilize a sinking fluid particle the system is essentially inverted. Design variables affecting the velocity profile in the observation section include the geometries of the conditioning elements and the divergent cone.

#### **Task 4. Chemical Sequestration**

OST's chemical sequestration portfolio is narrow and focuses upon one topic--mineral carbonation. This project attempts to form calcium and magnesium carbonates from minerals high in these metals, such as olivine and serpentine. Metal carbonates are desirable because they are benign, very stable and long-lived in the environment. Once these carbonate minerals are formed, they are appropriate for long-term unmonitored storage. Mineral carbonation can be defined as the reaction of  $\text{CO}_2$  with non-carbonate materials to form geologically stable mineral carbonates, such as calcite ( $\text{CaCO}_3$ ) or magnesite ( $\text{MgCO}_3$ ). Drawing on mineral carbonation to reduce  $\text{CO}_2$  emissions has a myriad of potential advantages. First, mineral carbonation mimics the natural weathering of rock. Mineral carbonates, the principal product of the process, are known to be stable over geological time (millions of years). For this reason, mineral carbonation ensures permanent fixation rather than temporary storage of  $\text{CO}_2$ . Second, Mg-rich silicates, especially serpentines, already exist in readily minable deposits/outcrops in quantities far in excess of that needed to carbonate all anthropogenic  $\text{CO}_2$  that could be emitted from the world's fossil fuel reserves. Finally, the silicate mineral carbonation reaction is strongly exothermic, providing clear energy and process cost advantages. NETL has been conducting a series of mineral carbonation tests at its Pittsburgh, PA facility over the past 2 years as part of a Mineral Carbonation Study Program within DOE. Other participants in this program include Los Alamos National Laboratory, Arizona State University, and Albany Research Center.

We have studied the effect of NaCl (i.e., 0.5 M  $\text{Na}_2\text{CO}_3$ /0.5 M  $\text{NaHCO}_3$ , and different concentrations of NaCl) on the extent of reaction utilizing an olivine sample, a non-hydrous magnesium silicate, from the Twin Sisters Range, Washington, USA. Each carbonation reaction was conducted using a continuously stirred tank reactor (CSTR) under identical conditions:  $T = 185^\circ \text{C}$ ;  $P_{\text{CO}_2} = 115 \text{ atm}$ ; time = 3 hours. The initial test was performed in a sodium carbonate/sodium bicarbonate only solution (0.5 M  $\text{Na}_2\text{CO}_3$ /0.5 M  $\text{NaHCO}_3$ ) whereas the two remaining tests were performed employing the identical sodium carbonate/sodium bicarbonate solution along with 1.0 M NaCl and 2.0 M NaCl additions, respectively.

The relationship between NaCl concentration and extent of the carbonation reaction is depicted graphically in Figure 4. As shown, the solutions containing 2.0 M NaCl and 1.0 M NaCl had increased yields of carbonated product. The yields of magnesite ( $\text{MgCO}_3$ ) were 80% for 2.0 M NaCl and 67% for 1.0 M NaCl. In comparison, the sodium carbonate/sodium bicarbonate solution only experiment produced a lower yield (i.e., 61%).

Mineral carbonation experiments utilizing a coal-derived Ca and Mg rich fly ash and a waste product obtained from the Dravo-Lime, Corporation, Pittsburgh, PA were also performed using a CSTR. Each carbonation test was conducted under identical conditions:  $T = 185^\circ \text{C}$ ;  $P_{\text{CO}_2} = 115 \text{ atm}$ ; time = 3 hours, solution = 0.5 M  $\text{Na}_2\text{CO}_3$ /0.5 M  $\text{NaHCO}_3$ ; 1.0 M NaCl. Duplicate carbonation experiments performed on the fly ash gave a 79% yield of carbonates, with calcite and dolomite being the principal products. Experiments conducted with the Dravo-Lime waste yielded a 50% conversion to calcite.

### **Task 5. Geological Sequestration Modeling**

Advances in high-speed computing and improved understanding of chemical behavior and fluid flow in porous media permit the use of simulations and modeling as tools for designing, optimizing, analyzing, and better understanding of chemical and physical processes. The Geological Sequestration Modeling task will integrate computational science capabilities within the Carbon Sequestration Science Focus Area, and build upon the solid foundation of experimental research at NETL. It will complement and support the laboratory and field work, and will promote a more thorough understanding of the fundamental science we are seeking to provide. The major emphasis of the OST laboratory effort in the Carbon Sequestration Science Focus Area is on geological sequestration and capture technologies. Accordingly, a complementary suite of computational science capabilities will be developed in these areas as well. In FY01 and in future years, a holistic approach (consisting of laboratory and modeling and simulation studies conducted in concert) to acquiring the fundamental body of knowledge required to successfully take carbon sequestration to fruition will be undertaken.

### **Task 6. Process Modeling and Economic Assessment**

Another essential element of this effort is an attempt to develop a thorough and accurate understanding of the costs and benefits of any new carbon dioxide separation and capture process technologies. In the Process Modeling and Economic Assessment task, evolutionary improvements to existing capture process designs will be sought, and the overall economics of various capture technologies will be evaluated. This work will be conducted outside of OST within the NETL's Process Engineering Division. NETL is developing models of existing carbon dioxide capture technologies such as those used in plants that employ monoethanolamine and the Selexol Process. Once modeled, the scientific and technological aspects of each process will be closely studied to determine if the most recent advances in science and technology could further improve their efficacy and economic viability if they were incorporated into the plant design.

### **Common Themes**

Investigation of the chemical interactions between  $\text{CO}_2$ /water/rock is a constant theme throughout the Carbon Sequestration Science Focus Area. It permeates the Geological Sequestration, Oceanic Sequestration and Chemical Sequestration tasks, and pervades the

Geological Modeling task. Developing a comprehensive understanding of the formation of Ca/MgCO<sub>3</sub> by the reaction of CO<sub>2</sub> with minerals, or CO<sub>2</sub> with water to form carbonate anion, bicarbonate anion, and carbonic acid, and their subsequent reactions with minerals or brine, either above or below ground, is vital to much of the work. The kinetics of these reactions must be better defined. The interactions of CO<sub>2</sub> and seawater (brine) to form hydrates is the major emphasis of the work in the Oceanic Sequestration task.

Another common theme is the development of facilities and capabilities to permit NETL to perform state-of-the-art Carbon Sequestration Science R&D. Three projects are concerned with improving NETL's facilities and capabilities. Specifically, we are undertaking two scoping exercises. The first is directed at finalizing the design of a versatile facility to investigate novel and modified techniques for CO<sub>2</sub> capture from fossil fuel processing. The second scoping exercise is to plan and design a state-of-the-art facility where geotechnical properties of candidate sequestration strata and chemical reactions between CO<sub>2</sub>, brine, oil, natural gas, coal, and associated strata can be investigated. The concept is to plan and design a research facility where geotechnical properties and chemical reactions can be investigated for a variety of geological formations into which CO<sub>2</sub> can be injected. The strategy is to produce a flexible Geological Sequestration Simulation Facility (GSSF). Similarly, NETL hopes to improve its ability to perform meaningful studies of sequestration in deep brine aquifers by acquiring brines and associated strata from five to ten potential deep aquifer sequestration sites scattered throughout the country. This will be performed in conjunction with the USGS.

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<b>Table 1. Variables of Interest</b>		
<b>Parameters</b>	<b>Values</b>	<b>Rationale</b>
pH	2	some thermal springs, water containing pyrite oxidation products
	6	bicarbonate buffered water
	9	sea water, high extreme
Salinity	0	Low extreme
	30 g/L NaCl	Seawater
Gas	CO <sub>2</sub>	Pure sample
	Combustion gas	“dirty” sample
Temperature(C)	15,25,35,45	Range of geologic sequestration, determination of binding energies
Pressure (atm)	1,50,100,150	Range for geologic sequestration

Figure 1. Typical TGA experiment (Batch #1 sorbent @ 80C absorption).

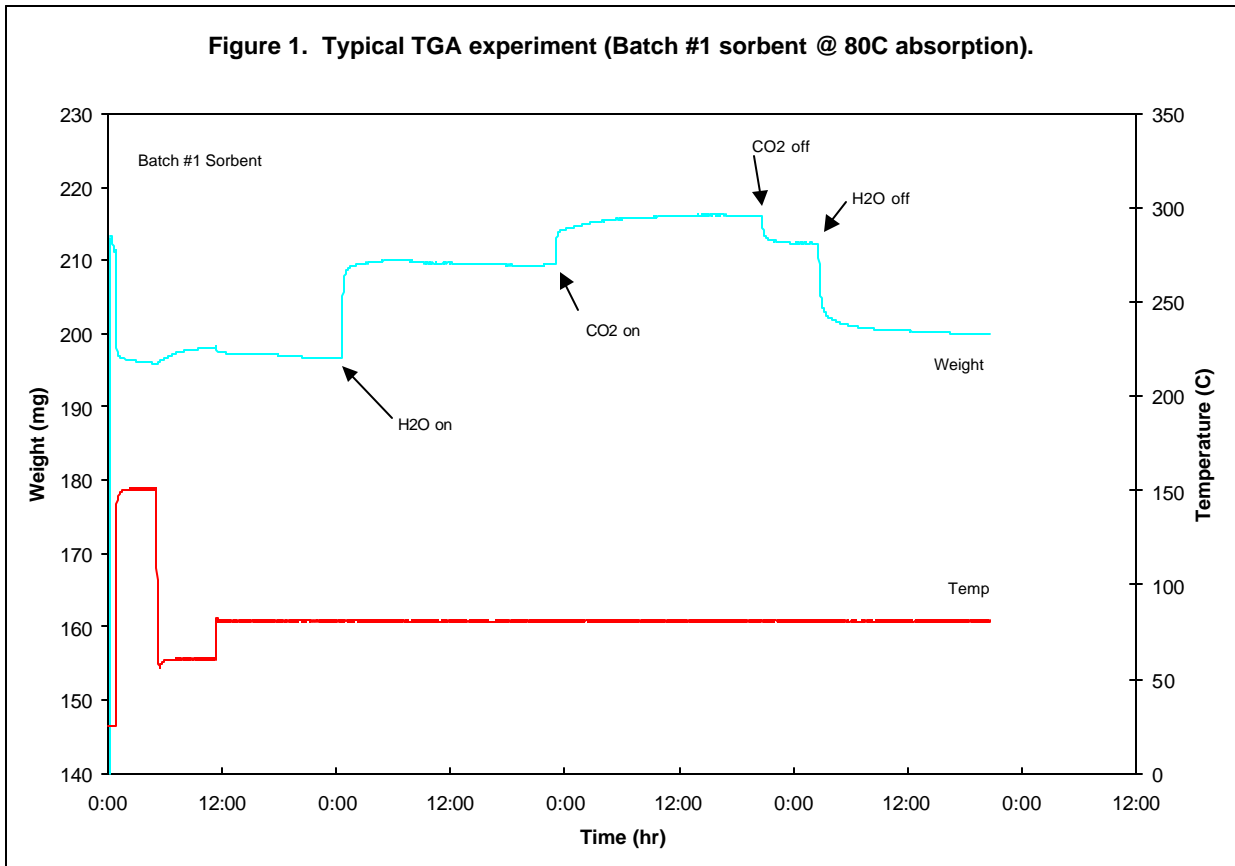
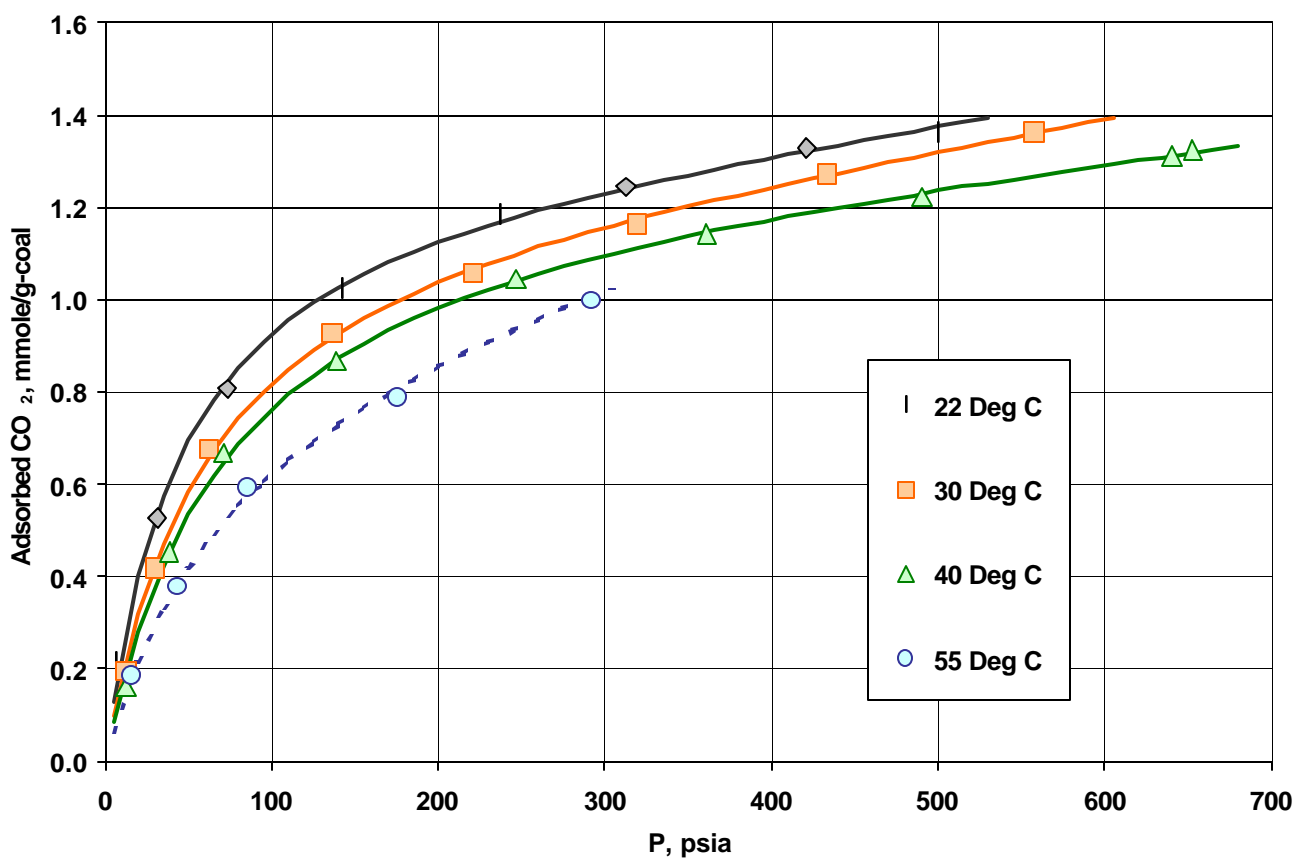
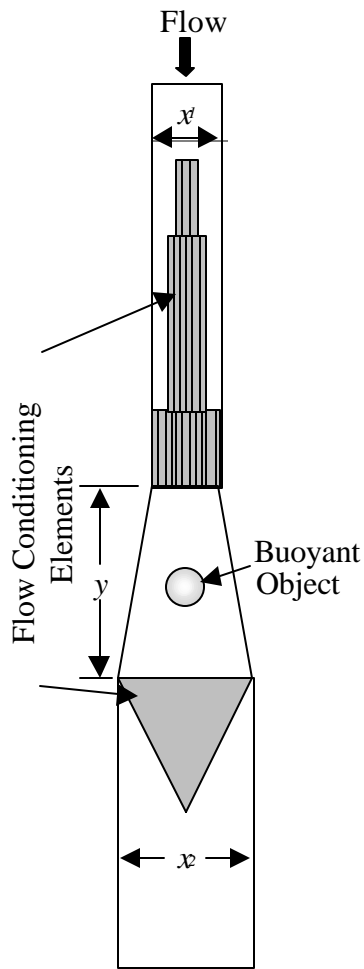


Figure 2. Temperature Effect on CO<sub>2</sub> Adsorption of Upper Freeport Coal





**Figure 3.** Schematic diagram of a water tunnel device

**Figure 4. Effect of NaCl on Conversion**

