A New Method for Tracing Seepage from CBNG Water Holding Ponds in the Powder River Basin, Wyoming
Final Report for a 2-year project (March 2008 – February 2010)
PIs: Shikha Sharma, K.J. Reddy, and Carol Frost

Abstract:

The proposed work will establish and verify the utility of a low-cost and innovative approach for understanding *"Groundwater contamination caused by seepage out of CBM water holding ponds,"* which has been identified as one of the critical areas of research under the CBNG Related Issues category in the Wyoming Water Research Program Request for Proposals (WRP RFP, 2008). Groundwater degradation caused by infiltration from CBNG water retention ponds is an issue of immense importance because groundwater is a major source for stock water, irrigation and drinking water for many small communities and ranchers in the Powder River Basin, Wyoming. It is necessary to develop a tracer that can fingerprint this water in order to trace seepage of water from these ponds into shallow aquifers. Strontium isotopes and other geochemical tracers have limited application in some instances because of significant contributions of these elements from local lithologies and high analysis costs. This study evaluates a low cost tracer that is less readily overwhelmed by near-surface sources.

Based upon preliminary analyses of CBNG co-produced water from the Powder River Basin, Wyoming, we suggest that the carbon concentrations and isotopic composition of Dissolved Inorganic Carbon ($\delta^{13}C_{DIC}$) can be used as a natural tracer for fingerprinting CBNG co-produced water. Our results show that CBNG co-produced water has strongly positive $\delta^{13}C_{DIC}$ (+12 to +22‰) that is readily distinguished from the negative $\delta^{13}C$ of most surface and groundwaters (-10 to -15‰). Furthermore, the DIC concentrations in co-produced water samples are also high (>100 mg C/l) compared to the 20-50 mg C/l in ambient surface and groundwaters of the region. The distinctively high $\delta^{13}C$ and DIC concentrations allow us to identify surface and groundwaters that have incorporated CBNG co-produced water and can also be used to track the CBNG produced water infiltrating from the ponds. Accordingly, we suggest that the $\delta^{13}C_{DIC}$ and DIC concentrations of water can be used for long term monitoring of infiltration of CBNG co-produced water from the CBNG water holding ponds (Sharma and Frost, 2008).

Samples will be collected from the CBNG discharge wells, water holding ponds and monitoring wells in the Powder River Basin and analyzed for $\delta^{13}C_{DIC}$ and DIC concentrations, pH, dissolved oxygen (DO), electrical conductivity (EC), major cations (e.g., Ca, Mg, Na, and K), and major anions (e.g., alkalinity, sulfate, chloride, fluoride, nitrate, and phosphate) to assess changes in water quality as the CBNG water migrates along the recharge flow path.

The results from this study will demonstrate how we can trace the seepage out of CBNG water holding ponds using a **low cost stable isotope approach**. A graduate student will be an integral part of this project. The project results will be presented at state, regional, and national meetings and published in appropriate peer-reviewed journals.

Objectives

Potential groundwater degradation caused by infiltration from CBNG water holding ponds is an issue of immense importance in the state of Wyoming where infiltration ponds are a common method for disposal of CBNG co-produced water. The objective of this study is to establish the utility of a new method for tracing infiltration of CBNG water from these ponds to near surface aquifers and shallow groundwaters. The method involves using stable isotope of carbon in dissolved inorganic carbon ($\delta^{13}C_{DIC}$) and DIC concentrations for fingerprinting CBNG co-produced water. The specific tasks which will be undertaken to attain this broad objective include:

1) The CBNG co-produced water samples will be collected from discharge points, corresponding retention ponds and a series of monitoring groundwater wells at selected sites in Powder River Basin for a period of 2 years.
2) Samples will be analyzed for $\delta^{13}C$ of DIC and DIC concentrations in water at the University of Wyoming Stable Isotope Facility.
3) Isotope mixing models will be used to calculate the fraction of CBNG co-produced water incorporated into the shallow groundwaters.
4) The geochemical parameters (pH, EC, major cations, major anions) will be used to assess the impact of infiltration of CBNG co-produced water on the ground water quality.
5) Convey research results to WY-DEQ, water users, landowners, and CBNG operators through project demonstrations, workshops, and local meetings.

Methods

Study sites have been chosen in Sheridan, Campbell and Johnson Counties (see Figure1). The sites were selected based on following criteria:

- All impoundment sites had upstream and downstream monitoring wells installed at similar depths (~40-140 feet) and in similar lithological horizons.
- All impoundments had received CBNG water for at least 1-2 years and had similar water holding capacity.
- Lithological logs were available for all monitoring wells.

The water sampling is done in accordance with the SAP (Sample Analysis Procedures) protocols of Wyoming DEQ-Water Quality Division. The monitoring wells are purged at rate of less than 1L /min with a submerged bladder pump until 3 casing volumes of water was removed. Water samples are collected when all field parameters (pH, EC, and Temperature) stabilized to within 10% for 3 consecutive readings. Three set of samples are collected at each sampling site 1) one sample for $\delta^{13}C_{DIC}$ and DIC concentration measurement 2) Duplicate samples for $\delta^{18}O$ and δD measurement and 3) Duplicate samples for alkalinity, major anions (phosphate, nitrate, fluoride, chloride, sulfate), and major cations (aluminum, boron, barium, cadmium, chromium, copper, iron, manganese molybdenum, lead, zinc, sodium, magnesium).

The samples for dissolved inorganic carbon are taken into a 60mm syringe and passed through a 0.45 μm Whatman filter attached to the end of the syringe and filled into a 30mL Wheaton glass serum vial. Two drops of benzalkonium chloride are added to halt biologic activity. The vials are topped with a Teflon seal and capped with an aluminum top. The aluminum tops are crimped to close the vials. The $\delta^{13}C_{DIC}$ is measured on a Gas Bench-II device

coupled to a Finnigan DELTA plus mass spectrometer in the central Stable Isotope Facility at the University of Wyoming. The reproducibility and accuracy was monitored by replicate analysis of internal lab standards and was better than ± 0.1 ‰. The $\delta^{13}C_{DIC}$ values are reported in per mil relative to V-PDB. The DIC concentrations in samples were also quantified from the mass spectrometry data. Three $NaHCO_3$ stock solutions of different DIC concentrations were prepared for this purpose. DIC concentrations were then quantified based on the peak areas of the mass 44-ion trace of these standards. Plotting peak area of CO_2 vs. concentration of DIC in these standards gives an excellent correlation (r^2=0.995), indicating that DIC concentrations of the samples could be quantified using this method. The relative standard uncertainty of the DIC concentration measurement in this study was ± 3%. The samples for $\delta^{18}O$ and δD are taken in 10 mL glass vials. The sample is filled up to the brim avoiding any headspace and then capped and sealed with layer of parafilm. The $\delta^{18}O$ and δD measurement is done using the Los Gatos Liquid-water Isotope Analyzer housed in University of Wyoming Stable isotope Facility. Samples for alkalinity, major anions and major cations are taken in Fisher 1L plastic bottles which had been previously acid washed for three hours in a nitric acid bath at a pH below 2. Upon return to Laramie, these samples are filtered with 0.45 μm filters before testing for alkalinity, anions (aluminum, boron, barium, cadmium, chromium, copper, iron, manganese, molybdenum, lead, zinc, calcium, potassium, and arsenic), and cations (fluoride, chloride, nitrate, phosphate, and sulfate). Alkalinity is tested in the University of Wyoming Water Quality Lab using the 702 SM Titrino automatic titrator manufactured by Brinkmann. Anions and cations are measured using the ICP-MS and IC housed in UW Soil Testing Laboratory and Geochemistry Analytical Laboratory respectively.

Principal Findings

Samples were collected from seven sites located in the Tongue and Powder River watersheds of the Powder River Basin (**Fig. 1**). At each samp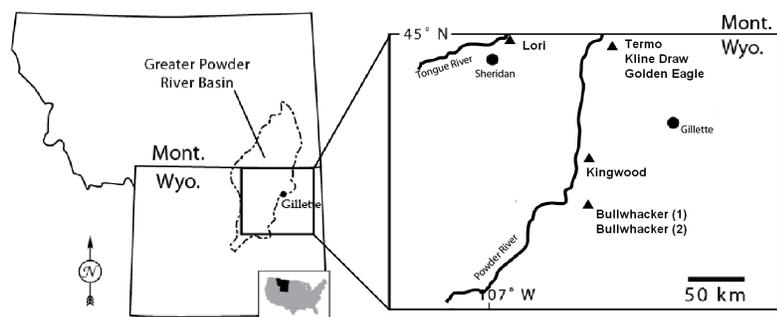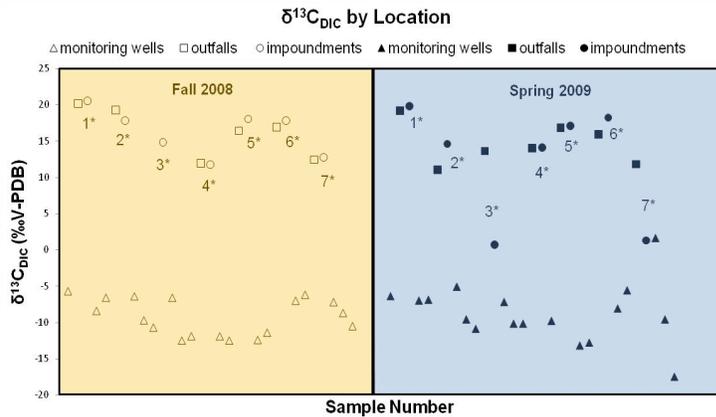ling location, we collected water from the outfall, impoundments into which this water is discharged, and samples from monitoring wells installed upstream and downstream of the impoundment. These study sites were chosen because they were instrumented with monitoring wells both upstream and downstream from the impoundment, had

Figure 1: Map showing location of sampling sites in Powder River Basin

received CBNG water for at least 2 years, and had a water holding capacity of at least 10 acre feet. In order to study the effects of seasonal changes, each location was sampled during the low flow season of September 2008 and again during the snowmelt season of May 2009.

Sampling trips were taken in August, September, and November of 2008, and May of 2009. The scientists from Wyoming Department of Environmental Quality office in Sheridan helped us sample four study sites namely Termo, Gloden Eagle, Kline Draw located in northwest corner of Campbell county and Lori located in the north-central Sheridan county of Powder river

basin (**Figure 1**). Sampling trips were taken to sample three sites, Kingwood, Bullwhacker (1) (P23-32-4376), and Bullwhacker (2) (P23-30-4376) in Johnson County with the assistance of WWC Engineering, an environmental consulting firm based in Sheridan. All the samples have been analyzed for $\delta^{13}C_{DIC}$ and, as hypothesized, all water samples collected from the outfalls and

*1 Kline Draw; 2 Termo; 3 Golden Eagle; 4 Kingwood; 5 Bullwhacker (1); 6 Bullwhacker (2); 7 Lori

Figure 2: Samples collected from ponds and outfalls have higher $\delta^{13}C_{DIC}$ values than water samples from all monitoring wells. During Spring, 2009, samples from Golden Eagle and Lori impoundments show much lower $\delta^{13}C_{DIC}$ values.

ponds have high $\delta^{13}C_{DIC}$ values in range of +12 to +20‰ during the fall and from to +1‰ to +18‰ durig the spring (**Figure 2**). The ponds and outfalls at all the sites had very similar $\delta^{13}C_{DIC}$ signatures during the fall, indicating that the pond water has not undergone any significant change in carbonate chemistry due to dissolution effects or due to CO_2 exchange with atmosphere. The monitoring wells had lower $\delta^{13}C_{DIC}$ values ranging from -13‰ (interpreted as not affected by seepage from ponds) to -6‰ (interpreted as effected by seepage). During the spring, some of the ponds show lower $\delta^{13}C_{DIC}$

signatures compared to the corresponding outfall. We hypothesize that during spring these impoundments may have been diluted by snowmelt water which has lower $\delta^{13}C_{DIC}$ signatures.

The carbon isotopic distinction between the two bodies of water (methanogenic impoundment water and non-methanogenic ambient water in monitoring well) is the basis for the

Figure 3: Plot of $\delta^{13}C_{DIC}$ against 1/DIC concentrations where the Y intercept represents the $\delta^{13}C$ signature of added DIC

use of $\delta^{13}C_{DIC}$ as a tracer. It can be expected that if methanogenic water from CBNG impoundments with high $\delta^{13}C_{DIC}$ values is infiltrating to the subsurface, it will elevate the $\delta^{13}C_{DIC}$ values of ambient groundwater. However, it is difficult to estimate the exact contribution of CBNG co-produced water using this approach mainly because there are several other contributors to the total DIC of sample like c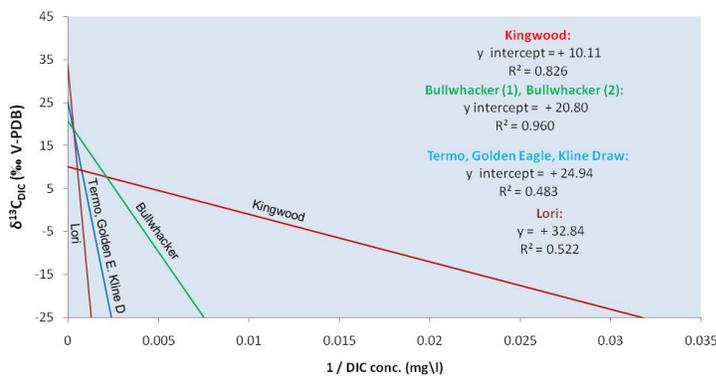arbon from carbonate dissolution, snowmelt, infiltrating surface waters etc. We used a Keeling plot approach to determine the major source of DIC. Using this approach the $\delta^{13}C_{DIC}$ values of samples are plotted against 1/DIC concentration and the regression of the data yields a y-intercept value, representing $\delta^{13}C_{DIC}$ of added DIC (**Figure 3**).

$$\delta^{13}C_{sample} \, (DIC)_{sample} = \delta^{13}C_{ambient} \, (DIC)_{ambient} + \delta^{13}C_{added} \, (DIC)_{added}$$

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$$\delta^{13}C_{sample} = \delta^{13}C_{ambient} \left(\delta^{13}C_{ambient} - \delta^{13}C_{added} \right) *1/(DIC)_{sample} + \delta^{13}C_{added}$$

Addition of DIC with $\delta^{13}C$ values >10 ‰ is strong evidence that bicarbonate originating from biogenic methanogenic processes is the major source of DIC at these sites rather than carbonate rock dissolution which contributes DIC with $\delta^{13}C$ values of ~1-2 ‰. Another line of evidence supporting that carbonate dissolution is not a major source of DIC to the waters in monitoring wells is that samples from monitoring wells showing higher $\delta^{13}C_{DIC}$ values (indicated by higher % contribution from methanogenic waters using our $\delta^{13}C$ mixing model) do not necessarily show higher Ca concentrations (Figure 4). Therefore, we can presume that the higher $\delta^{13}C_{DIC}$ values in these monitoring well samples are due to contribution from methanogenic water with higher $\delta^{13}C$ signatures.

Assuming that the co-produced water infiltrating from the impoundment and the ambient water in the sampled aquifer at the monitoring well site are the only two sources contributing to the total DIC, a simple two end member isotope mixing model can be used to estimate the fraction of CBNG co-produced water incorporated into the groundwaters at the monitoring well sites:

$$\delta^{13}C_{mw} \, (DIC)_{mw} = \delta^{13}C_{iw} \, f_{iw} \, (DIC)_{iw} + \delta^{13}C_{amb} \, (1- f_{iw}) \, (DIC)_{amb}$$

Where the subscripts "mw", "iw", and "amb" indicate the carbon isotope ratio ($\delta^{13}C$), fractional contribution (f), or DIC concentration (DIC) of monitoring wells, impoundment and ambient water samples, respectively.

*The upstream monitoring well at Lori was not included because it is located beneath a CENG co-produced water irrigated field. .

Figure 4: Two end member mixing model for fall, 2008 samples. Percent contribution from the impoundment is shown by gray bars. Calcium concentrations depicted by solid black squares connected by the black line. Note the wells showing higher percent contribution from impoundments (i.e. wells with higher $\delta^{13}C_{DIC}$ values) do not necessarily have higher Ca concentrations.

Ambient $\delta^{13}C_{DIC}$ values were considered to be the lowest $\delta^{13}C_{DIC}$ value obtained from the monitoring wells in each study area. During fall 2008, ambient water $\delta^{13}C_{DIC}$ values were -10.7‰ at Kline Draw, Termo, and Golden Eagle; -12.5‰ at Bullwhacker (1), and Bullwhacker (2); -12.5‰ at Kingwood; and -10.5‰ at Lori. All of these samples have $\delta^{13}C_{DIC}$ values within the expected isotopic range of ambient groundwaters (Mook and Tan, 1991; Sharma and Frost, 2008). The total concentration of DIC for each sample was calculated from the addition of the concentrations of carbonate, bicarbonate, and carbonic acid species.

Results from the isotope mixing model from fall 2008 data suggest that both the upstream and downstream monitoring wells can be influenced from infiltration of CBNG co-produced water from the impoundments. The fraction of contribution from CBNG impoundments ranges from 0-10.4% in upstream wells to 0-14.1% in downstream wells **(Figure 4)**. We did not use this model for samples collected in spring 2009 because there was excessive dilution of impoundment water with isotopically light snowmelt water significantly lowering the $\delta^{13}C_{DIC}$ values of the CBNG water in the impoundments at the Golden Eagle and Lori sites. This dilution reduced the isotopic range between the two end-members of the mixing model, the impoundment water (iw) and water in the monitoring well (mw), resulting in an exaggeration of calculated fractional contribution of impoundment water. We would like to point out that this simple two member mixing model approach has several limitations: 1) it does not account for other potential sources of DIC to the water in the monitoring well, e.g. dissolution of carbonates as water percolates through the different lithological horizons, 2) it does not account for any other source of water to the monitoring well, such as snow melt recharge or water seeping in from overlying or underlying aquifers, and 3) it does not account for uncertainty in the estimated proportions due to changing isotopic composition of the two end-members during different seasons, years, etc.
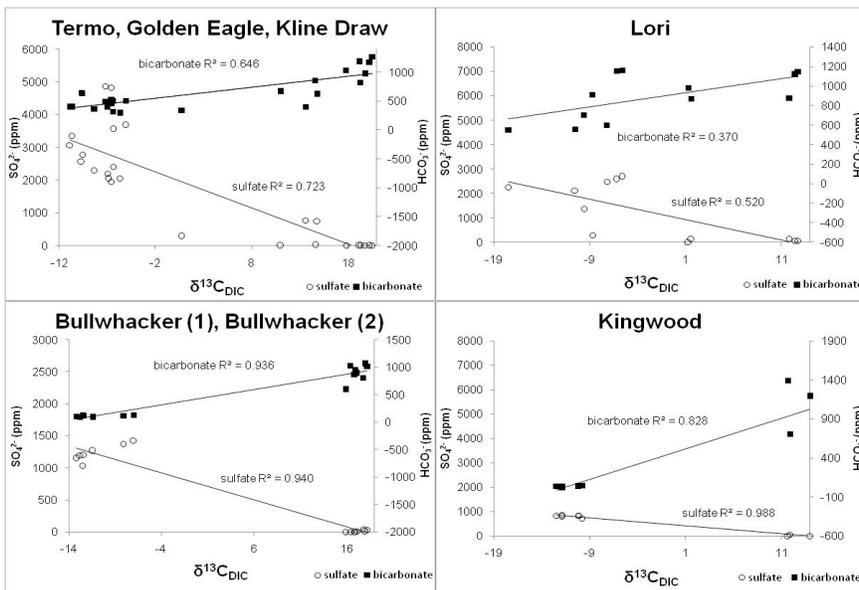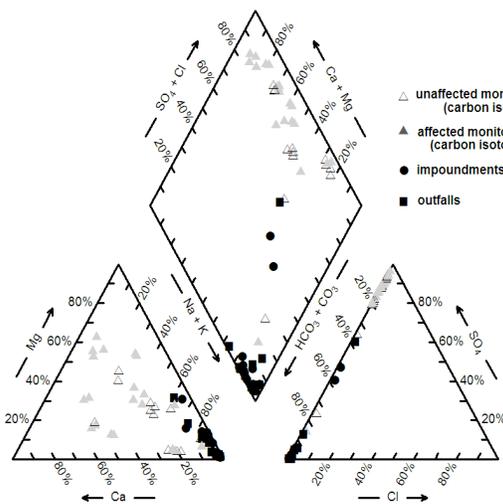

The major anion chemistry of the water samples indicates that the methanogenic waters with high $\delta^{13}C_{DIC}$ values have low concentrations of sulfate and high concentrations of bicarbonate (**Figure 5**). The low SO_4^{2-} and high HCO_3^{-} concentrations in CBNG co-produced waters are probably the result of bacterially-mediated oxidation–reduction reactions in the coal zones (Van Voast 2003; Rice et al., 2008; Brinck et al., 2008). To compare major ion content of co-produced water

Figure 5: Graph showing positive correlation of $\delta^{13}C_{DIC}$ with HCO_3 concentration (○) and negative correlation with SO_4 (#) concentrations

from outfall and impoundments to monitoring wells geochemical variation piper diagrams were made with AqQaChem water analysis spreadsheet software from RockWare. Outfall and

impoundment water samples are predominantly of the Na-HCO₃- type as has been documented by previous studies (Van Voast, 2003; Patz et al., 2004; Brinck and Frost, 2007; Jackson and Reddy, 2007; Brinck et al., 2008; Healy et al., 2008; Rice et al, 2008). The samples from monitoring wells are highly variable probably due to site to site variation in local lithologies. However, most of these samples are SO_4^{2-} dominant. There is no clear geochemical difference between wells that have received some contribution of methanogenic water based on the carbon isotope proxy model, and those that have not. The TDS (Total Dissolved Solids) values in the waters of the monitoring wells is generally higher than methanogenic waters from outfalls and impoundments and could be the result of series of dissolution reactions which take place as the water recharging these deep aquifers infiltrates through the various lithologies (Wheaton and Brown, 2005; Frost and Brink, 2005; Brinck and Frost 2007). The average TDS values of samples collected from both seasons from wells that have received some CBNG discharge are very similar to wells that show no indication of infiltration based on out carbon isotope model. This suggests that geochemical processes that occur during infiltration are the most important contributor to shallow groundwater TDS (Brinck and Frost, 2007) or that co-produced water influence in affected monitoring wells is not significant enough to alter the TDS. The SAR (Sodium Absorption Ratio) is higher in methanogenic waters due to higher Na concentrations mainly because the high HCO₃ concentration causes all the Ca and Mg to precipitate as carbonate.

	TDS mg/L	SAR	SO₄ mg/L	HCO₃ mg/L	Mg mg/L	Ca mg/L	Na mg/L
Termo, Golden Eagle, Kline Draw							
Outfalls and Impoundments	1970	25.2	167	1272	28	16	475
Affected Monitoring Wells (5.0%)*	5335	4.7	2993	652	492	411	580
Unaffected Monitoring Wells	5326	6.9	2950	783	351	417	744
Kingwood							
Outfalls and Impoundments	2055	18.9	27	1472	24	10	480
Affected Monitoring Wells (0.4%)*	1378	9.5	854	49	11	92	360
Unaffected Monitoring Wells	1303	9.3	798	67	9	83	335
Bullwhacker (1), Bullwhacker (2)							
Outfalls and Impoundments	1908	13.4	11	1352	34	16	413
Affected Monitoring Wells (2.4%)*	2095	2.9	1270	163	84	333	223
Unaffected Monitoring Wells	2285	2.5	1039	195	60	280	180
Lori**							
Outfalls and Impoundments	2235	42.2	72	1536	5	9	578
Affected Monitoring Wells (4.7%)*	3514	14.7	1320	1091	142	93	812
Unaffected Monitoring Wells	3627	11.1	1510	1012	147	177	735

*The average percent contribution of impoundment water to monitoring wells, based on the carbon isotope two end-member mixing model.
**The upstream monitoring well, Lori PD-1, was not included because it was drilled into methanogenic coal.

Where: SAR = (Na²⁺ meq/L) / √ [(Ca²⁺ meq/L) + (Mg²⁺ meq/L) / 2]

Legend:
△ unaffected monitoring wells (carbon isotope proxy)
▲ affected monitoring wells (carbon isotope proxy)
● impoundments
■ outfalls

Figure 6 Piper diagram comparing impoundment, outfall, and monitoring well water samples from fall 2008 and spring 2009. Monitoring wells are indicated as being affected or unaffected by CBNG co-produced water based on the carbon isotope/dissolved inorganic proxy. The table shows average TDS, SAR , SO_4^{2-} , HCO_3^{-}, Ca, Mg and Na values of samples collected from both seasons (spring 2008 and fall 2009) for each study site.

We hypothesize the as CBNG co-produced water infiltrates through the different lithologies concentrations of Ca and Mg increase due to dissolution resulting in the decrease of SAR values. This is likely because calcite, dolomite and gypsum are the common minerals in shallow soil and bedrock profile of the Powder River Basin and their dissolution increases the Ca, Mg and SO4 ion concentrations in infiltrating waters decreasing the SAR (Wheaton and Brown, 2005). However no difference is seen in wells affected and not affected by impoundment seepage.

However, in this study the highest impacted monitoring well shows only 14% contribution from CBNG co-produced water. Continued monitoring is required to understand how the geochemical parameters of these well waters change as the fractional contribution from CBNG co-produced water increases over time.

This project also partially supported another study entitled *"Stable Isotope and Geochemical Analyses of Wyoming Coalbed Aquifers: A new tool to minimize water production and maximize gas production in a coalbed natural gas play"*. This is a MS thesis project of Scott Quillinan graduate student in department of Ge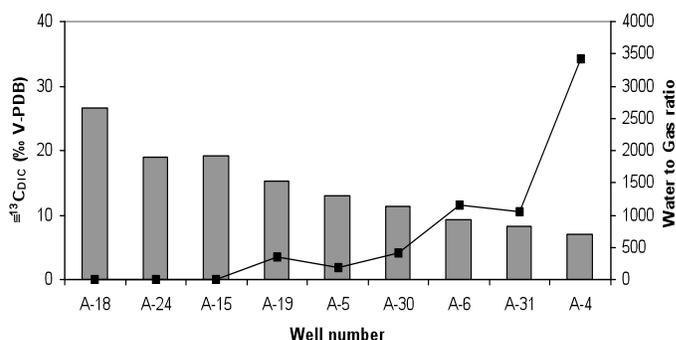ology and Geophysics, co-advised by Drs. Frost and Sharma. The primary funding sources of this project are Anadarko Petroleum and Wyoming Geological Survey. However, partial financial support in terms of stable isotope sample analysis and sampling supplies was provided by this WWDC-USGS funded project. In this study we are investigating isotopic and water chemistry over a wide variety of attributes in the Atlantic Rim and Powder River Basin CBNG plays. Preliminary samples have been collected and geospatial modeling of the data

Figure7: The well-head water samples showing low $\delta^{13}C_{DIC}$ values had high water to gas ratio indicating water being withdrawn from overlying and underlying aquifers during de-watering process.

collected so far indicates that the $\delta^{13}C_{DIC}$ signatures used in conjunction with geochemical data and geological information can be effectively used for 1) CBM reservoir characterization 2) coal zone distinction and 3) test for hydraulic isolation and well completion efficiency. In Atlantic Rim area the carbon isotope signatures of CBNG co-produced waters shows a good correlation with water/gas ratios i.e. higher $\delta^{13}C_{DIC}$ signatures are correlated with low water/gas ratios. A set of sample samples collected from well-heads located in same coal zone (name not disclosed due to proprietary issues) and located in close proximity showed highly variable $\delta^{13}C_{DIC}$ values **(Figure 7)**. We found that well-head water samples (collected from same age wells) having a very low $\delta^{13}C_{DIC}$ value had very high water to gas ratios indicating that during the process of de-watering significant amount of water was being withdrawn from overlying and/or underlying aquifers at these sites. The initial results indicate that the $\delta^{13}C_{DIC}$ signatures of co-produced waters could potentially be used as an exploratory tool for planning locations of future expansion of CBNG development in areas where gas production can be maximized with minimum water production. Another set of preliminary samples were collected from CBNG wells completed in Big George Coal in Powder River basin and they show some very interesting trends. The high $\delta^{13}C_{DIC}$ values correlate with low water/gas ratios in the south-eastern part of the study area. However, in the central and some portions of the northwestern corner of the study area low $\delta^{13}C_{DIC}$ values correlate with low water to gas ratios. In the central part of the basin some of this anomaly overlaps with the enormously thick zone of the Big George Coal Seam. We hypothesize that fresh recharge of nutrient rich water or contributions from thermogenic pathways could be

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partly responsible for this isotopic excursion. Scott is currently in process of interpreting the data and writing his thesis.

Significance

The results from this project indicate that methanogenic waters in the study area can be distinguished from ambient groundwaters by their higher Na and HCO_3^-, and relatively lower Ca, Mg and SO_4^{2-} concentrations. However, site to site variations in bedrock/soil and water chemical composition results in wide range of values for these parameters limiting their applicability as effective tracers of CBNG co-produced waters. The higher $\delta^{13}C$ of DIC of co-produced CBNG water on the other hand can prove to be an effective tracer to trace seepage out of CBNG water holding impoundments. Preliminary results from water samples collected from well-heads of producing wells in Atlantic Rim and Powder River Basin indicate strong correlations between enriched $\delta^{13}C_{DIC}$ and water to gas ratios. The carbon isotope technique developed by these two projects can potentially help in addressing regulatory issues related to discharge of CBNG co-produced water and also help CBNG operators to maximize gas production while maintaining optimal drilling costs and protecting the valuable groundwater resources of the region.

Student Support Information

Graduate : Josh Baggett MS student in Renewable Resources advised by Dr. Sharma is working on this project for his Masters thesis in Rangeland Ecology and Watershed Management/ Water resources. He received training in taking field water quality measurements, geochemical and isotope sampling protocols, geochemical analysis and stable isotope analysis.

Scott A. Quillinan MS student in Department of Geology and Geophysics co-advised by Drs. Frost and Sharma received training in taking field water quality measurements, geochemical and isotope sampling protocols, geochemical analysis and stable isotope analysis.

Undergraduate : Paul Haselhorst, a Geology major and technician of Dr. Sharma, received training in preparing samples for geochemical and isotopic analysis and also in running samples on the stable isotope ratio mass spectrometer.
Patrick Warden technician of Dr. Sharma and Biology major received training in preparing and running isotopic analysis on water samples.

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- Quillinan S., Frost C.D and Sharma S. (in prep) Carbon isotope technique for coalbed aquifer characterization; Powder River Basin, Wyoming. AAPG Bulletin.
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