



Laboratory and field analysis of flowback water from gas shales



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ABSTRACT

Flowback water is usually highly saline and the salt concentration varies by time and well location. Understanding the origin of the flowback salts is essential for evaluating fracturing and flowback processes. In this study, laboratory and field analyses are performed to investigate the origin of the flowback salts. The field data includes the total salt concentration (salinity), individual ion concentration, pH, and dissolved oxygen measured during the flowback process for three wells completed in the Horn River Basin. The rock mineralogy is determined using XRD. The cation exchange capacity (CEC) of shale samples are measured using ammonium acetate method. Water and oil imbibition experiments are conducted for shale samples of different surface-to-volume ratio. The individual ion concentration is measured during the water imbibition experiments using ICP-MS and IC. EDXS analysis is used to investigate the surface of natural fractures.

Noticeable amount of barium found on the surface of natural fractures suggests that the barium in the flowback water primarily originates from the natural fractures. Furthermore, the samples with higher clay content have higher CEC. During the water imbibition process, these samples have higher and faster ion transfer from shale-to-water; suggesting the mobilization of the exchangeable ions from the clays. During the water imbibition experiment, the Na/Cl and K/Cl ratios are initially high and decrease at the later times. Leaching of the exchangeable sodium and potassium ions from the clay minerals is a possible reason for the initial high Na/Cl and K/Cl molar ratios. The dissolution of chloride-bearing components increases the chloride concentration, which decreases the Na/Cl and K/Cl molar ratios at later times. The measured pH is slightly above 8 for all of the flowback water samples. The presence of natural buffer systems such as calcite and dolomite may explain the neutral pH range of the flowback water.

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1. Introduction

Low permeability shale gas reservoirs are rapidly emerging as a vital source of natural gas (Frantz and Jochen, 2005). Multi-lateral horizontal drilling and multi-stage hydraulic fracturing technologies have paved the way for economical exploitation of these shale resources. In hydraulic fracturing, a large volume of fracturing fluid (mainly water) is injected into the reservoir to create multiple fractures and increase reservoir contact per well (King, 2012). Then, the wells are sometimes shut-in for a period of time (soaking period) (King, 2012; Lan et al., 2014a; Makhanov et al., 2014). During the production phase, a portion of the injected fracturing fluid returns to the surface, i.e. flowback water, alongside the produced hydrocarbons (Dehghanpour et al., 2012, 2013; King, 2012; Abbasi et al., 2012; Bai et al., 2015).

The field data indicate that the chemistry of flowback water is substantially different than that of the injected water (Rimassa et al., 2009; Haluszczak et al., 2013; Bearinger, 2013; Zolfaghari et al., 2015a, 2014; Engle and Rowan, 2014; Capo et al., 2014). For instance, in the Horn River Basin (HRB), slick water (which has similar salinity levels as fresh water) is injected into the reservoir to create fractures (Johnson and Johnson, 2012), while the recovered flowback water is highly saline (40,000–70,000 ppm) (Bearinger, 2013; Zolfaghari et al., 2015a; Engle and Rowan, 2014; Capo et al., 2014).

Several past studies focused on the flowback chemical analysis to evaluate the hydraulic fracturing operations and characterize the fractured reservoir. Chemical analysis models for optimizing fracturing fluid cleanup have been developed by Woodroof et al. (2003), Sullivan et al. (2004), and Asadi et al. (2006). Gdanski (2010) incorporated a chemistry layer to a 2-D numerical simulator to history match the composition of the flowback fluid. Engle and Rowan (2014) applied a multivariate data analysis approach to investigate the geochemical evolution of flowback water

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Nomenclature

A_{sp}	specific surface area	HRB	Horn River Basin
$A_{sp,high}$	high specific surface area	IC	ion chromatography
$A_{sp,low}$	low specific surface area	ICP-MS	inductively coupled plasma mass spectrometry
$A_{sp,medium}$	medium specific surface area	Mu	Muskwa
CEC	cation exchange capacity	OP	Otter-Park
DI	deionized	XRD	X-ray diffraction
EC	electrical conductivity	θ	contact angle
EDXS	energy-dispersive X-ray spectroscopy		
Ev	Evie		

(Engle and Rowan, 2014). Flowback salt concentration data from hydraulically fractured wells in the HRB were analyzed by Ghanbari et al. (2013); and their results indicate that the architecture of the induced fracture network affects the load recovery and the shape of the salt concentration profiles. Bearinger (2013) developed a qualitative theory explaining the relationship between shape of the salt concentration profile and the complexity of the fracture network. In our previous study (Zolfaghari et al., 2015a), we presented a model to mathematically describe the Bearinger's qualitative theory. This model characterizes the complexity of the fracture network using the salinity profile measured during the flowback process.

Despite the previous studies that analyze the chemistry of flowback water to assess the hydraulic fracturing operations, the origin of flowback salts is still a matter of dispute. Blaunich et al. (2009) reported that the production of salts during the flowback process is due to the dissolution of shale constituents in injected water; while Haluszczak et al. (2013) claimed that the saline flowback water is mainly caused by interaction of injected water with the in situ formation brine. Furthermore, shales are composed of clay minerals (Carman and Lant, 2010) that can alter the chemistry of the water through ion exchange reactions. Moreover, shales have mineral-filled natural fractures (Gale et al., 2014). Reactivation of these mineral-filled natural fractures and dissolution of minerals into the water during hydraulic fracturing operations is another possible source for flowback salts.

In this study, a laboratory and field analysis is performed to investigate the origin of salts in the flowback water. The salinity and individual ion concentration are measured for three wells completed in the HRB. X-ray diffraction (XRD) analysis is utilized to characterize the mineralogy of the rock samples. Cation exchange capacity (CEC) of the shale samples are measured using ammonium acetate method. Oil and water imbibition experiments are performed on samples of three different surface-to-volume ratios (i.e. specific area or " A_{sp} ") to investigate the effects of fluid-rock interface on the liquid uptake and diffusion rate of individual ions. Electrical conductivity (EC) and individual ion concentration are measured during the water imbibition process. Furthermore, elemental maps from energy-dispersive X-ray spectroscopy (EDXS) analysis are used to visualize the distribution of individual ions on the fresh break and on the natural fracture surfaces of the samples.

2. Methodology

Interactions of injected water with in situ formation brine and shale rock are the possible sources of the flowback salts. Since the shale formations in the HRB are at sub-irreducible water saturation state (Dehghanpour et al., 2013), there is a possibility for ions to precipitate in the form of salts in the pore structure or on the surface of natural fractures. To identify the origin of the

flowback water salts, we first analyze the field flowback data including salinity and individual ion concentration. The elemental maps of the fresh break and natural fracture surfaces from EDXS analysis are compared to identify the nature of the locally occurring minerals. The EDXS results are further compared with the bulk-rock mineralogy, CEC, and the flowback water chemistry to identify the source of the ions.

2.1. Field data

The flowback water and downhole shale samples are collected from three wells completed in the Muskwa (Mu), Otter-Park (OP), and Evie (Ev) formations in the HRB. Fig. 1 shows the location of the target wells in the HRB.

The salinity of the flowback water is frequently measured during the flowback process. Fig. 2 shows the field data of flowback water salinity versus time for three target wells. The salinity profiles of Mu and OP initially show a gradual increase, and then reach a plateau at around 40,000 ppm; the salinity profile of Ev continues to increase even after 70,000 ppm.

The individual ion concentrations are measured during the flowback process for the three target wells. Fig. 3 shows the concentration trends for barium, iron, sulfate, and chloride ions measured during the flowback process.

Over the course of the flowback process, a general increase of the barium and sulfate concentration is observed for all three wells. A similar increase in the concentration of chloride ion is also observed for the Mu and Ev wells (no data was available for the OP well). The iron and sulfate concentration profiles are relatively scattered. The concentration of iron ions in OP shows a gradual increase over the first 200 h during the flowback process, after which the concentration decreases with time.

2.2. Laboratory analysis

2.2.1. Bulk-rock mineralogy

One of the current hypotheses about the origin of the flowback salts is that the salts in the flowback water are the shale constituents that have been dissolved in the injected water (Blaunich et al., 2009). XRD analysis is applied to characterize the rock mineralogy of the shale samples.

2.2.2. Contact angle measurement

During fracturing operation water imbibe into the rock matrix. The water present in the pore network provides a medium for ion transfer into/out of the rock. Thus, the rock affinity for water uptake impacts the ion transfer between the rock and water. On the other hand, the wettability of shale rock impacts the liquid uptake by shales (Xu and Dehghanpour, 2014; Lan et al., 2014b). Fluid-rock contact angle is a simple way to characterize the wettability of the shales (Andrew et al., 2014; Mirchi et al., 2015). So, in

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