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Evaluation of anionic and non-ionic surfactant performance for Montney shale gas hydraulic fracturing fluids

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Abstract

Hydraulic fracturing is often used in unconventional shale reservoirs, and 50%–95% of the injected hydraulic fracturing fluid remains in the formation due to the capillary effect. This phenomenon has been observed in the Montney shale formation, Canada, where the flowback water recovery is generally less than 25%. Surfactant is one of the hydraulic fracturing fluid additives for reducing surface tension and capillary forces to facilitate water flowback recovery. Surfactant loss due to adsorption by the reservoir rocks reduces the chemical's efficiency, and this causes water retention in the formation and reduces water flowback recovery. The compatibility of surfactant with reservoir rock is critical to minimize surfactant adsorption on the rock surface because this diminishes the primary function of the surfactant hydraulic fracturing fluid additive and to ensure cost-effectiveness. This study evaluates surfactant efficiency to improve flowback recovery for the Montney shale formation based on IFT, surface tension, and adsorption. This study evaluates surfactant performance and performs a fluid–fluid interaction experiment and fluid-rock compatibility investigation. Several commercial surfactants are screened for low interfacial tension and surface tension. Further analysis is carried out by evaluating the fluid-rock compatibility using the static soaking test at reservoir pressure and temperature. The pre-soaking and post-soaking test fluids were analyzed for water composition, liquid–liquid interfacial tension, surface tension, and pH. Results showed that the selected surfactant is a critical determiner of the hydraulic fracturing fluid performance. SOLOTERRA 938 is an anionic surfactant that has good compatibility with Montney shale formation. Unlike other non-ionic surfactants, SOLOTERRA 938 retains the interfacial tension and surface tension after seven days of interaction with reservoir rock. The interfacial tension remained unchanged at 0.1 mN/m. The surface tension decreased from 28.4 to 27.5 mN/m with air and from 21.7 to 20.8 mN/m with hydrocarbon because surfactant behavior changes with pH change. The surfactant concentration was measured using high-pressure liquid chromatography, and the loss was 12% after seven days of interaction with the reservoir rock (from 0.1 to 0.088wt%). The adsorption calculated based on the concentration volume showed a low value of between 0.43 and 0.97 mg/g rock.

Keywords Hydraulic fracturing fluids · Surfactant · Adsorption · Fluid-rock compatibility · Shale formation · Flowback water

Introduction

The hydraulic fracturing technique was first used in 1947 in the Hugoton field, Kansas (Barati and Liang 2014). This technique is used in a low permeability reservoir to stimulate the reservoir by creating a conductive fracture network to facilitate the flow of fluids from the formation to the wellbore (Gharibi and Zoveidavianpoor 2015). A large volume of hydraulic fracturing fluids and proppant is pumped at high pressure and flow rate into the wellbore. The pumping fractured the formation when the wellbore pressure exceeded the fracture initiation pressure (Li et al. 2013).

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The hydraulic fracturing fluid injected into a shale gas reservoir consists of a hydraulic fracturing base fluid, proppant, and a mixture of low concentration chemical additives such as a surfactant, friction reducer, biocide, breaker, iron control, and scale inhibitor (Ferrer and Thurman 2015). The additive used and its concentration are dependent on the water and formation properties. Generally, hydraulic fracturing fluids consist of approximately around 99% mass of based fluid and proppant along with 1% mass chemical additives (Heyob et al. 2017). Figure 1 shows the hydraulic fracturing fluid additives.

Flowback water is one of the major concerns in a hydraulic fracturing operation. According to Chai et al. (2019), 50% to 95% of the injected hydraulic fracturing fluid remained in the formation due to the capillary effect. The flowback water recovery ranges from 10 to 60% (Ghanbari and Dehghanpour 2016; Lu et al. 2018; Chai et al. 2019). The invading water can hinder hydrocarbon flow from the formation to the fractures due to water blockage and eventually impairs well productivity (Liang et al. 2020). Therefore, improved flowback recovery could enhance hydrocarbon production.

In field operations, water blockage is mitigated by shutting in the well or using surfactant as fracturing fluid additives. The well shut-in requires a long delay of hydrocarbon production, which could trigger clay swelling because of the reservoir rock exposure to fracturing fluid (Liang et al. 2020). Surfactant is used as fracturing fluid additive to reduce surface tension, lower the capillary forces to facilitate the recovery of the injected fluid, alter wettability, and reduce flow friction (Penny et al. 2005; Kaufman et al. 2008; Fichter et al. 2010; Ferrer and Thurman 2015; Mirchi et al. 2015; Yue et al. 2016; Chai et al. 2019; Rabie et at. 2019). It also reduces the interfacial tension between the injected hydraulic fracturing fluid and targeted formation. The hydraulic fracturing fluid requires a low interfacial tension to enter the induced hydraulic fracture and pre-existing natural fractures (Fichter et al. 2010).

He et al. (2015) compared the performance of nonsurfactant fluid, weakly emulsion surfactant fluid, and non-emulsion surfactant fluid in shale reservoirs. Weakly emulsion surfactant fluid yielded higher oil recovery relative to the non-surfactant fluid. The enhancement in surface clean-up efficiency and productivity is better relative to non-emulsion surfactant (He et al. 2015). Liang et al. (2020) reported the same observation where water trapping occurred in the no surfactant case.

The surfactant may lose its effectiveness due to adsorption and cause water retention in the formation matrix and reduces water flowback recovery (Fichter et al. 2010; Rabie et al. 2019). The hydraulic fracturing fluid that remained in the formation may cause water blockage and reduce well productivity (Rabie et al. 2019). Lu et al. (2018) reported that the main reason for hydraulic fracturing fluids retention in the formation and reduced water recovery was surfactant adsorption on the newly fractured micro-fracture surfaces. A more developed fracture network is more likely to retain water in the formation.

The compatibility of the chemicals and formation is critical for an effective hydraulic fracturing in reservoir stimulation. However, the interaction mechanism between the hydraulic fracturing fluid additive and reservoir rock is not fully understood because shale formations have different mineralogy and lithology. Each shale formation requires a specific hydraulic fracturing fluid additive recipe. According to Sun (2014), the design and selection of hydraulic fracturing fluid additives are dependent on the formation properties; however, there are no comprehensive criteria for hydraulic fracturing fluid selection (Sun 2014). The incompatibility between hydraulic fracturing fluids additives and reservoir rock can cause reservoir chemical damage.

Surfactants can be adsorbed on the formation surface and significantly alter the surface properties and cause phase trapping (Fichter et al. 2010). It can reduce the interfacial tension between water and the formation. Surfactants can also change the contact angle of the fluid with the rock surface and alter the change the fluid wettability of the formation. This facilitates recovery, especially in a tight gas reservoir (Penny, Pursley and Holcomb, 2005). Viscoelastic

Fig. 1 Hydraulic Fracturing Fluids Additives (Ferrer and Thurman, 2015)
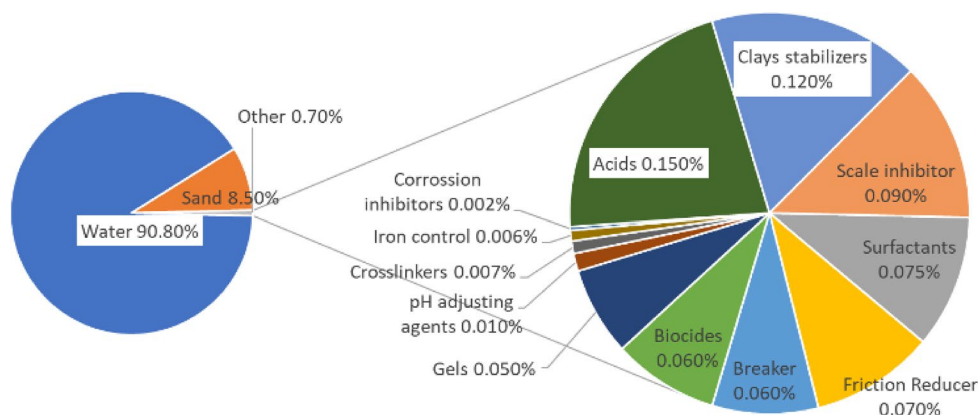
(VES) surfactant is generally used in hydraulic fracturing because its polymeric behavior reduces the friction during high-pressure fluid pumping.

Three types of viscoelastic surfactants are used in hydraulic fracturing fluids, cationic, anionic, and non-ionic surfactants. The cationic surfactants head carries a positive charge which may be permanent or only exists in a certain pH range (Rapp 2017). The adsorption of surfactants is influenced by the rock surface-fluid interface charge (Liu et al. 2004). Cationic surfactants are easily adsorbed and precipitated if the rock surface has a negative charge (Zhang et al. 2010). Anionic surfactant head carries a negative charge and exhibits high solubility in hydrocarbon but low solubility in aqueous solution (Fink 2013). The adsorption of anionic surfactant in shale or sandstone is low or even negligible due to repulsive electrostatic interaction with most natural surfaces since many natural surfaces are negatively charged under natural condition (Muherei and Junin 2009; Amirianshoja et al. 2013). Non-ionic surfactant head does not carry a charge (Belhaj et al. 2020). Muherei and Junin (2009) reported that non-ionic surfactants are more prone to adsorption on shale than sandstone because of hydrogen bonding and hydrophobic bonding. It was reported that the quantity of non-ionic surfactant adsorbed increased as the percentage of clay minerals increased (Amirianshoja et al. 2013).

Fluid adsorption and absorption influence the estimated hydrocarbon volumes that may occur in unconventional plays. Relative to conventional reservoirs, unconventional resources are characterized by small pores and large surface areas (Smith and Terracina 2006). Consequently, adsorption and absorption play more critical roles in unconventional resources than conventional reservoirs. Adsorption is the adhesion of molecules on solid surfaces. Absorption causes the molecules to form the bulk phase of the solid. However, in porous materials such as shale and kerogen, the difference between absorption and adsorption may not be apparent. It

is difficult to determine whether molecules adhere to the pore wall or reside within the pore volume when the pore diameter is comparable to molecular diameters. This paper focuses on the concept of adsorption, although adsorption and absorption are not readily distinguishable in some cases.

Materials and methodology

Recycle-produced water/flowback water and preparation of the testing fluid

Recycle-produced water is the water from recovered flowback water that was collected in multiple ponds from the field as shown in Fig. 2. The water was delivered to laboratory using cold box to preserve and reduce composition change. The recycle-produced water was filtered with 100 mesh screens to mimic the field filtration level and the water composition, and the results are listed in Table 1. The water was then mixed with different type of surfactant using magnetic stirrer for 1 h. The solution was referred as surfactant hydraulic fracturing fluid.

Surfactant

Table 2 lists the 22 screen surfactants, which comprise 13 non-ionic, four anionic, two Zwitterionic, and two unknown head-type surfactants. The surfactants were obtained from chemical vendor suppliers. They have different purity range and were used as it is without any purification. The surfactants were screened for IFT < 0.1 mN/m and surface tension < 28mN/m. IFT and surface tension key performance was based on the trends identified from a chemical company survey. The screened surfactants selected for this study, SOLOTERRA 938, GlucoPure Wet, and Aspiro S2420X, are of the anionic and non-ionic types.
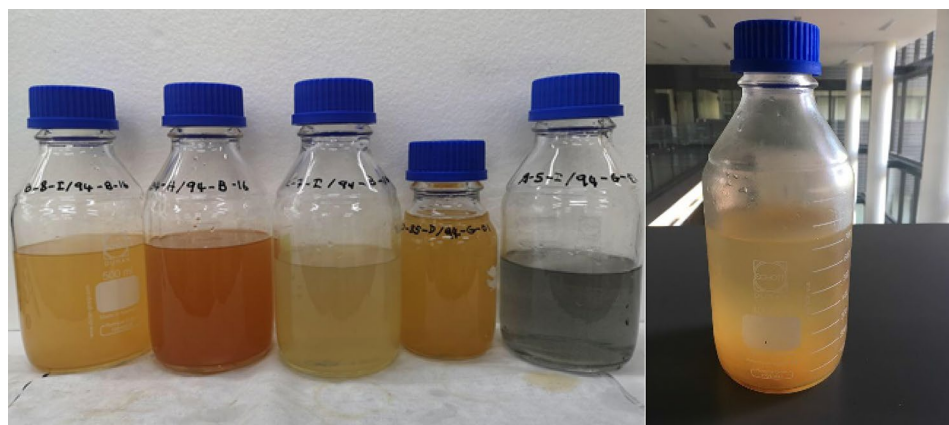

Fig. 2 Recycled-Produced Water as Received

Table 1 Water Composition

No	Test Description	Method	Unit	Result
1	Temperature	APHA 4500 H + ,B	deg C	24.7
2	pH	APHA 4500 H + ,B	-	6.16
3	Density	ASTM 4052#	Kg/m3	1.11606
4	Conductivity	APHA 2510B#	µS/cm	182,100
5	Salinity	APHA 2520B#	mg/L	over range
6	Total Dissolved Solid	APHA 2540C	mg/L	136,900
7	Boron,B	APHA 3120B	mg/L	13.76
8	Bismuth,Bi	APHA 3120B#	mg/L	<0.001
9	Lithium,Li	APHA 3120B	mg/L	55
10	Manganese,Mn	APHA 3120B	mg/L	<0.007
11	Phosphorus,P	APHA 3120B#	mg/L	0.49
12	Tin,Sn	APHA 3120B	mg/L	0.228
13	Free Chlorine	HACH 8167#	mg/L	0.12
14	Ammonia (as N)	HACH 8038/8155/8171#	mg/L	over range
	Anion			
15	Sulfate	HACH 8051#	mg/L	1
16	Chloride	APHA 4500Cl-B	mg/L	102,805
17	Bicarbonate, HCO3-	APHA 2320B	mg CaCO3/L	76.42
	Total Anion			102,882
	Cation			
18	Sodium,Na	APHA 3120B	mg/L	5122
19	Calcium,Ca	APHA 3120B	mg/L	7836
20	Magnesium,Mg	APHA 3120B	mg/L	837
21	Potassium,K	APHA 3120B	mg/L	1215
22	Iron,Fe	APHA 3120B	mg/L	0
23	Strontium,Sr	APHA 3120B	mg/L	1525
24	Barium,Ba	APHA 3120B	mg/L	241
	Total Cation			16,776

Shortlisted surfactant

SOLOTERRA 938 SOLOTERRA 938 is fatty alcohol poly-alkylene glycol ether acetic acid. It is an anionic surfactant and has an HLB of 12 that shows typical properties of non-ionic surfactant in the acid environment. SOLOTERRA 938 is neutralized with alkali to form chemical corresponding to ether carboxylates, and it is 69% active, ether carboxylic acid, sodium ether carboxylate Cn–(PO)z–(EO)y–COONa is expected to exhibit viscoelastic surfactant (VES) properties. The structure is shown in Fig. 3. As the characteristic of the brine system for fracturing fluid contains 150,000 TDS, it is ideal to use SOLOTERRA 938 as VES fracturing fluid without drag-reducing polymer at reservoir temperature.

GlucoPure wet GlucoPure® Wet is a low foam non-ionic surfactant with HLB of 13 with the active ingredient of 47% to 53% of sugar surfactant from the glucamide family, which is a non-EO solubilizer for hydrophobic R = C8/10 methyl ester with higher wetting capacity. GlucoPure® Wet composition is N-C8/10-acyl-N-methyl-glucamin, the structure as shown in Fig. 4. GlucoPure® Wet is miscible with all types of surfactants (anionic, non-ionic, cationic, and amphoteric). GlucoPure is chemically stable in acidic and alkaline media in the pH range of approximately 3 to 10.

Aspiro S2420X Aspiro S2420X is an alkyl polyglycosides (APG) and the molecule structure as shown in Fig. 5. APG is a non-ionic surfactant that would make form sources from renewable raw materials or natural base stocks such as vegetable oils and starch or other carbohydrate-based materials (Nickel et al. 2007; Han et al. 2015).

Hydrocarbon gas composition

Table 3 presents the composition of the hydrocarbon gas used in this study. It is an average gas composition data from the study wells in Montney formation.

Table 2 Twenty-two screened surfactants

No	Surfactant Name	Head Type (Non-ionic, Cationic, Anionic, Zwitterionic)	Chemical Name
1	SOLOTERRA 938	Anionic	ethoxylated carboxylate, Isotridecanol, ethoxylated, carboxymethylated
2	GlucoPure Wet	Non-ionic	Glucamine, N-C8/10-acyl-N-methyl-glucamin
3	Aspiro S2420X	Non-ionic	Alkyl polyglycosides, D-Glucopyranose, oligomeric, C10-16 alkyl glycosides
4	Lansurf OA14	Non-ionic	Oleic Acid + 14EO (PEG600 Monooleate)
5	Tergitol ™ NP-10	Non-ionic	Poly(oxy-1,2-ethanediyl), alpha-(4-nonylphenyl)-omega-hydroxy-, Polyethylene glycol, Dinonylphenyl polyoxyethylene, 10 EO mole
6	Tergitol ™ NP-9	Non-ionic	Poly(oxy-1,2-ethanediyl), alpha-(4-nonylphenyl)-omega-hydroxy-, Polyethylene glycol, Dinonylphenyl polyoxyethylene, 9 EO mole
7	Genapol EP 2552	Non-ionic	C12-15 OXO alcohol PO-EO adduct
8	SOLOTERRA 970	Anionic	Isotrideccan-1–01, ethoxylated, propoxylated, carboxymethylated
9	SOLOTERRA 963	Anionic	Alcohols, C16-17 branched and linear, ethoxylated, carboxymethylated
10	Tergitol ™ NP-7.5	Non-ionic	Poly(oxy-1,2-ethanediyl), alpha-(4-nonylphenyl)-omega-hydroxy-, Polyethylene glycol, Dinonylphenyl polyoxyethylene, 7.5 EO mole
11	Euroquat PK47	Zwitterionic	Cocamidopropyl betaine based on palm kernel oil
12	NOVEL® TDA-3 Ethoxylate	Non-ionic	Ethoxylated Alcohol, Trideceth-3. Derived from branched C13 alcohol and ethoxylated to an average of 3 mol of ethylene oxide
13	Plexsurf 200E		Not available
14	CRS 2080		Not available
15	Genapol EP 0244	Non-ionic	C10-C12 alcohol EO-PO adduct
16	Tergitol ™ NP-6	Non-ionic	Poly(oxy-1,2-ethanediyl), alpha-(4-nonylphenyl)-omega-hydroxy-, Polyethylene glycol, Dinonylphenyl polyoxyethylene, 6 EO mole
17	Euroquat HC47 VG	Zwitterionic	Cocamidopropyl betaine based on coconut oil
18	Tergitol ™ NP-8	Non-ionic	Poly(oxy-1,2-ethanediyl), alpha-(4-nonylphenyl)-omega-hydroxy-, Polyethylene glycol, Dinonylphenyl polyoxyethylene, 8 EO mole
19	Tergitol ™ NP-4	Non-ionic	Poly(oxy-1,2-ethanediyl), alpha-(4-nonylphenyl)-omega-hydroxy-, Polyethylene glycol, Dinonylphenyl polyoxyethylene, 4 EO mole
20	Lansurf OA10	Non-ionic	Oleic Acid + 10EO (PEG400 Monooleate)
21	Tergitol ™ NP-40	Non-ionic	Poly(oxy-1,2-ethanediyl), alpha-(4-nonylphenyl)-omega-hydroxy-, water, Polyethylene glycol, Dinonylphenyl polyoxyethylene, 40 EO mole
22	SOLOTERRA 961	Anionic	Alcohols, C16-18, ethoxylated, propoxylated, carboxymethylated
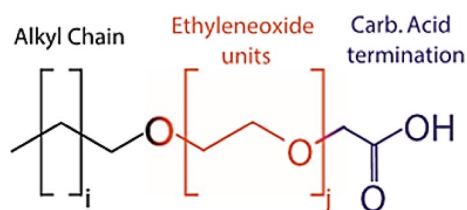
Fig. 3 Schematic Representation the Chemical Formula of Polyethylene Alkyl Ether Carboxylic Acids Composed of a Hydrophobic, Alkyl Chain, Hydrophilic Ethylene Oxide Units and Terminated by a Carboxy-Methyl Unit (Chiappisi, 2017)
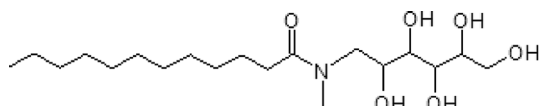
Fig. 4 Structure of Glucamine molecule
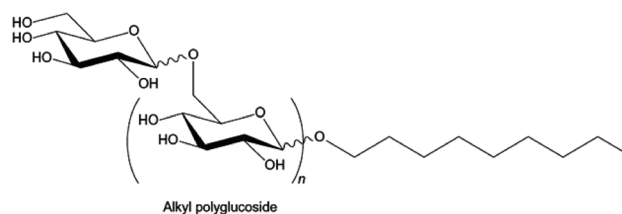
Fig. 5 Structure of APG molecule

Preparation and characterization of core samples

This study investigated the interaction between hydraulic fracturing fluids and three types of lithofacies. Table 4 lists the nine samples of 1-inch diameter used in the testing. The core samples are the native core samples obtained from Montney formation. The air method was employed to plug the core samples to minimize sample damage that may occur

Table 3 Hydrocarbon Gas Composition

Component		Mole %
Hydrogen	H2	0.07
Nitrogen	N2	0.17
Carbon Dioxide	CO2	0.23
Methane	C1	85.91
Ethane	C2	8.57
Propane	C3	2.76
i-Butane	iC4	0.64
n-Butane	nC4	0.77
i-Pentane	iC5	0.28
n-Pentane	nC5	0.21
Hexane	C6	0.20
Heptane	C7	0.19

Table 4 Core Availability by Lithofacies

Lithofacies	Number of core samples for Soaking Test
1 Sandy F–C Siltstone to Silty VF Sandstone	3
2 Bituminous F–C Siltstone	3
3 Bituminous F-M Siltstone	3
Total Number of Samples	9

Fig. 6 Philip CT scan

Fig. 7 Shimadzu X-ray Diffractometer Machine

due to the high clay content in shale. The core sample selection was based on the CT scan images and the supporting data from X-ray diffraction (XRD) and scanning electron microscopy (SEM). The samples were taken from a depth between 1999.87 and 2175.7 m. The sample permeability ranges from 14.71 to 1198.71 nanodarcy, and the porosity ranges from 0.12% to 6.97%. The core sample diameter ranges from 2.53 cm to 2.57 cm, and the core length ranges between 5.74 cm and 8.83 cm.

Core CT scan

Core CT scans measures the radiodensity of the core material at a sub-millimeter resolution. The dataset is presented as static 2D images or video scans (Hall and Govert 2016). CT enables detection and evaluation of internal geological structures such as bedding planes, fractures and nodules, lithological changes, and bulk density. This method is widely used in the industry for core selection since it provides insight into the core without exposing the core surface.

The CT scan used in this study is Philips Ingenuity CT Scan (serial number: 336233) as shown in Fig. 6. It is a computerized tomography (CT) or computerized axial tomography (CAT) scan that combines the data from several

X-rays to produce a detailed image of the structures inside the sample.

The nine core plug samples were subjected to core plug CT scan to evaluate the core state (heterogeneity, nodules, fractured, etc.). The results were used to select the core samples for routine analysis and special tests and avoid the selection of unsuitable sections. The CT scan was carried out with two axial scans and two transverse scans.

X-ray diffraction (XRD)

This study used X-ray diffraction (XRD) spectroscopy to analyze the rock samples from the Montney shale formation and determine their mineral composition. Figure 7 shows a

Shimadzu X-ray diffractometer machine (model XRD-700) used to perform the analysis.

The sample for the bulk rock XRD analysis was a representative portion of the sample, and about 10 g of the rock is used in routine analysis. The samples do not require preservation other than keeping them in ambient condition after sampling. The sample selected for XRD was dried, disaggregated, and cleaned of hydrocarbons using chloroform and methanol. The sample was gently ground using ceramic mortar and pestle to obtain a powder with 5–10 microns particle size. The powdered sample was packed in an aluminum holder and X-rayed from 2 thetas (θ) of 2 to 65 degrees. The mineral (i.e., peak) identification of the analyzed sample was performed at d-spacing of the detected peak in the X-ray diffractogram of the sample. The semi-quantitative method established by Huggett (1992) was employed to analyze the bulk mineralogical content (Huggett 1992). The peak intensity (either area count or height) for each mineral was multiplied with a specific factor, which was derived by measuring peak intensities of the standard minerals from known mixtures, and the quantification was derived using the Siroquant software.

High-pressure mercury injection

The AutoPore IV Series Automated Mercury Porosimeter was used to inject the high-pressure mercury. The mercury injection test was performed on trimmed ends samples. Mercury was injected at incrementally increasing pressure of up to 60,000 psi. The data were used to generate capillary pressure (Pc) curves and pore (throat) size distribution data. The high-pressure data reveal the extent of the microporosity.

Three point bending

Before the soaking test, the core samples were fractured using a three-point bending apparatus to obtain a larger contact surface and maximize the fluid-rock interaction. A minimal load was applied to the core samples to obtain a straight-line fracture across the core sample diameter. Figure 8 shows the three-point bending apparatus and its accessories, including the cutting blade and sample holder.

The cutting blade and sample holder are specially fabricated for this study, where the thinner cutting blade produced a thinner slick fracture. The holder prevents the samples from fracturing in other directions.

Surface tension measurement

This study used the pendant drop tensiometer (Vinci IFT700) to measure the gas–liquid surface tension. It is used to measure the IFT with values greater than 10^{-3} N/m. The measurement principle was based on the fluid droplet in a gas environment, where the fluid and gas are immiscible (Stauffer 1965; Moiré et al. 2016). A fluid droplet is attached to the end of a needle, and its profile is influenced by the IFT and density difference between the fluid and gas. A numerical analysis of the droplet profile and the Young–Laplace equations is used to determine the IFT (Moiré et al. 2016).

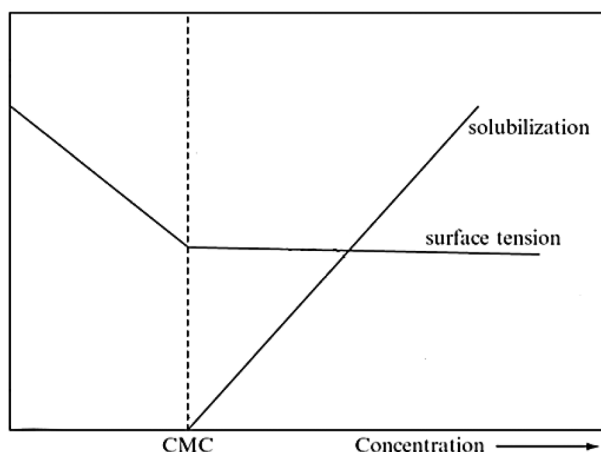**Fig. 8** Three-point Bending Testing Apparatus and Its Accessories
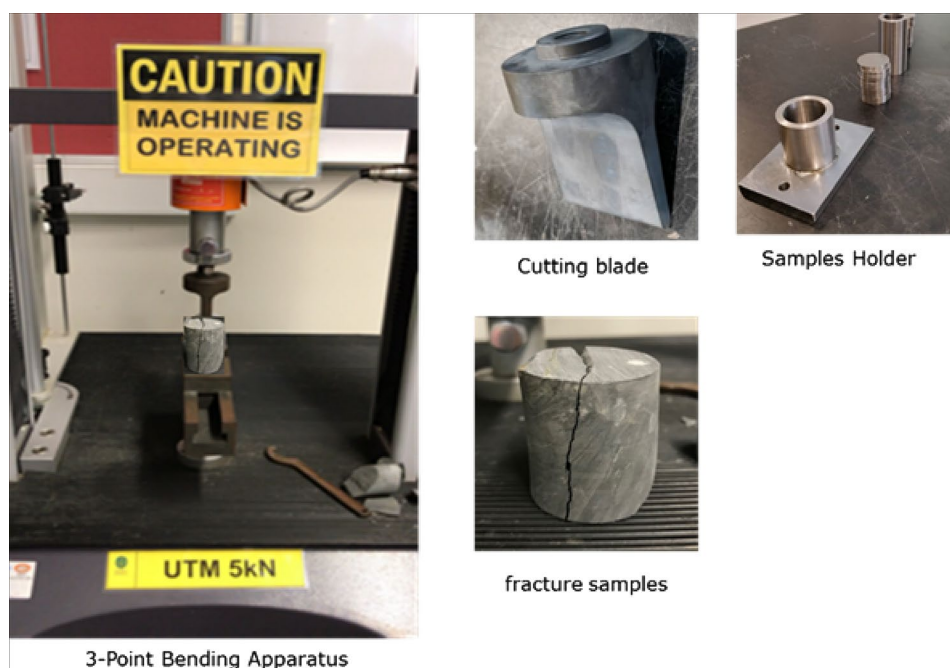
Fig. 9 Schematic representation of the concentration dependence of surface tension and solubilization properties for solutions of surfactant (Tadros, 2010)

Fig. 10 SOLOTERRA 938 Surface Tension with Concentration Relationship

The measurement was taken at room condition of 22 °C and 14.7 psia.

The surface tension technique was used to determine the critical micelle concentration (CMC) values. The surface tension values were plotted against surfactant concentrations to obtain the CMC, which is the lowest surface tension reading, and remains relatively constant after that point, as shown in Fig. 9.

The CMC of each surfactant varies and is influenced by several factors (Lu 2016).

- An increase in the surfactant's alkyl chain length will result in a considerably lower CMC.
- Generally, non-ionic surfactants have lower CMCs than the ionic surfactants with the same hydrophobic chain.
- Cationic surfactants typically have slightly higher CMCs than anionic surfactants.

GlucoPure® Wet is a low-foam non-ionic sugar-based surfactant with a chemical formula N-C8/10-acyl-N-methyl-glucamine. Its CMC is 0.0068 wt% at a temperature of 70 °C with methanol and water solution (Warwel et al. 2001). Aspiro S2420X is an APG-type non-ionic surfactant and has a CMC of 0.056 wt% for carbon chain C8 to C10 at 60 °C with distilled water (Nickel et al. 2007; Sulek and Wasilewski 2009).

The CMC for SOLOTERRA 938 was measured at eight different concentrations of 0.025, 0.05, 0.1, 0.2, 0.3, 0.4 and 0.5 wt%. Figure 10 shows that the CMC of SOLOTERRA 938 is at 0.1 wt%.

The CMCs for the surfactants range from 0.0068 to 0.1 wt%. SOLOTERRA 938 was measured using Montney
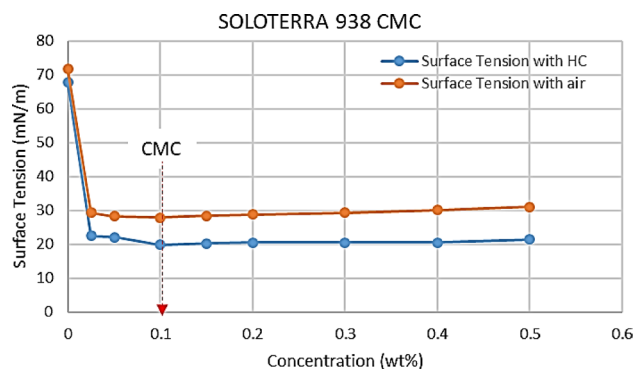
fluids and had the highest CMC. All soaking test surfactant was tested at 0.1 wt% concentration.

Therefore, the CMC value for 3 surfactants ranges from 0.0068 to 0.1 wt%. SOLOTERRA 938 was measured using Montney fluids, and given the highest CMC, all soaking test surfactant was tested at the concentration of 0.1 wt%.

Interfacial tension measurement

The equipment for measuring IFT is the spinning drop tension meter (Grace GM6500). The target IFT value for surfactant-based hydraulic fracturing fluids is 0.1 mN/m (millinewton/meter) and below. The measurement was taken using hydraulic fracturing fluid and condensate so that the condensate oil droplet can be easily displaced because of the resulting high capillary number (Wu et al. 2010). The measurement was taken at ambient temperature and pressure because the sample behavior is sensitive to pressure and temperature changes. This method involved injecting a droplet of Fluid 2 into a capillary tube filled with Fluid 1 (Vonnegut 1942; Moiré et al. 2016). The capillary rotation causes the injected droplet to elongate. The droplet profile is the result of the equilibrium between the centrifugal and interfacial forces. As with the pendant drop method, the IFT was determined using the numerical analysis of the droplet profile and the Young–Laplace equations. For an IFT less than 10^{-5} N/m, the measurement uncertainty may exceed 100% (Moiré et al. 2016).

pH measurement

The basic principle of electrometric pH measurement is determining hydrogen ions activity. The potentiometric measurement employed the standard hydrogen electrode and a reference electrode. The pH meter used in this study is from Mettler Toledo (seven compact model).

Soaking test

The fluid-rock interaction was initiated by performing the soaking test at reservoir pressure and temperature. Soaking is similar to leaving the well shut-in for an extended period after the flowback stage before putting it on production (Yaich et al. 2015). However, there is some concern with leaving hydraulic fracturing fluid in the well as it could cause formation damage. Yaich et al. (2015) investigated the impact of extended shut-in in different areas of the Marcellus shale and found that the well shut-in after flowback resulted in potentially higher initial gas flow rate and improved performance.

The soaking test was performed on the core samples from different lithofacies of the siltstone formation. The fluid and core samples weights in the soaking experiment were adapted from the soaking experiment for a limestone reservoir with some modification, such as the apparatus used. The core sample weight to hydraulic fracturing fluid weight ratio for the test is 1:4 (Sterpenich et al. 2009). The samples were soaked for seven days at reservoir pressure and temperature. A seven-day soaking is a minimum period before a hydraulic fracturing well start to flowback in Montney formation. Figure 11 shows that the system was pressurized using hydrocarbon gas to mimic the reservoir condition. A laboratory soaking experiment should represent the natural systems as much as possible (Sterpenich et al. 2009).

Analysis of water chemistry was carried out before and after soaking. After the soaking, the fluid was analyzed for water chemistry, and the core sample was analyzed using the high-pressure mercury injection (HPMI) (Fig. 12). The pre-soaking and post-soaking measurements include:

1. Liquid–liquid interfacial tension (IFT).
2. Surface tension.
3. pH.
4. Surfactant concentration and adsorption.

Surfactant concentration and adsorption

Surfactant concentration was measured using the high-performance liquid chromatography (HPLC) analysis. HPLC is a component separation technique using column chromatography that pumps a sample in a mobile phase (solvent) at high pressure through a column with a stationary phase. HPLC is used to determine the concentration of active components such as ether carboxylate, alkyl glucamine, and ethoxylated nonylphenol before and after the soaking tests. HPLC (Agilent 1100 Series, Agilent Technologies, USA) is equipped with an evaporative light scattering emission (ELSD) detector. Different reversed-phase columns were employed to separate the active components depending on the active ingredients of the materials. The mobile phase is methanol:water (v/v) in gradient flow. The column temperature differs depending on the surfactant type, and the injected volume is 5 µL. The external calibration based on the peak areas was used to calculate active ingredient concentration.

Fluid adsorption and absorption influence the estimated hydrocarbon volumes that may occur in unconventional plays. Unconventional resources are characterized by small pores and large surface areas (Smith and Terracina 2006), and adsorption and absorption play more critical roles in unconventional resources. Adsorption is the adhesion of molecules to solid surfaces. Absorption causes the formation
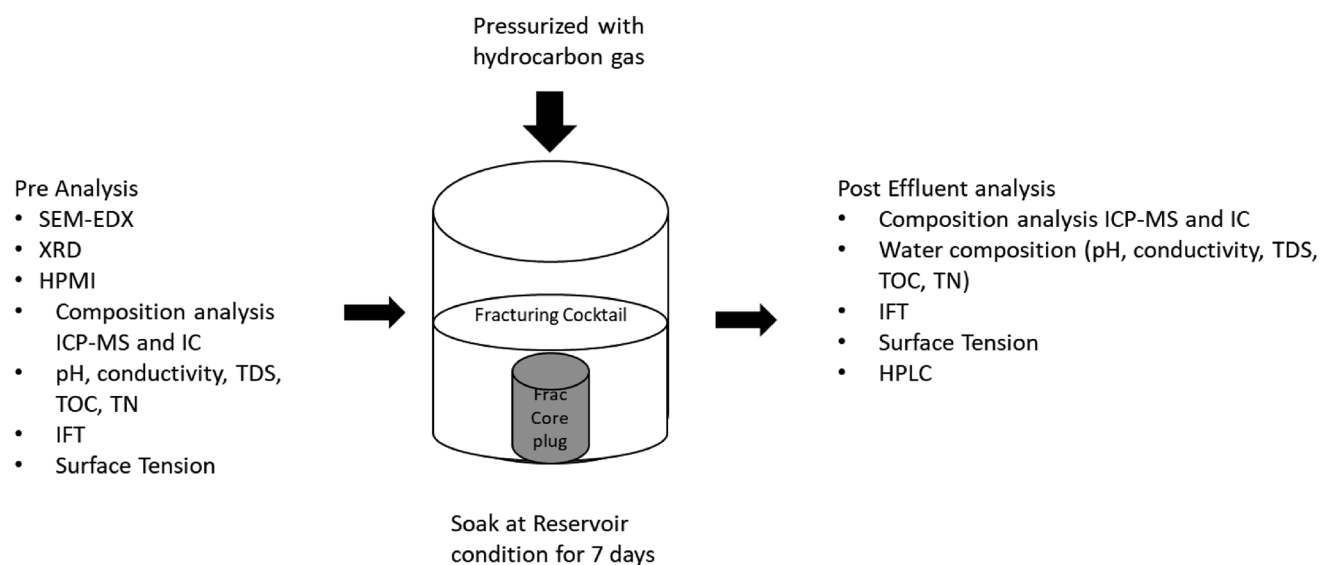

Fig. 11 Soaking test schematic diagram
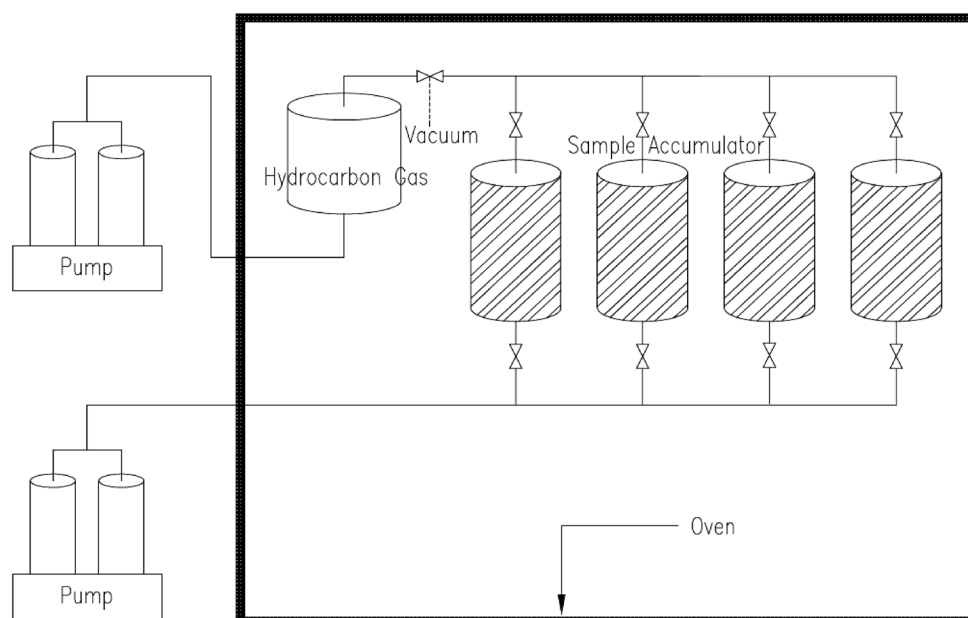
Fig. 12 Soaking test analysis workflow

of molecules in the bulk phase of the solid. However, the difference between absorption and adsorption may not be apparent in porous materials such as shale and kerogen. When pore diameters are comparable to molecular diameters, it is difficult to determine whether the molecules adhere to the pore wall or are within the pore volume. This section focuses on the concept of adsorption, although adsorption and absorption are not readily distinguishable in some cases.

A static adsorption experiment was carried out to quantify the loss of surfactant by adsorption onto a rock under simulated (static) conditions. Rock clay content, rock mineralogy, temperature, and salinity affect surfactant adsorption on rock surfaces. The static adsorption test used the fluid from the soaking test, and the test was performed on different lithofacies of siltstone reservoir in Montney formation.

The chemical adsorption unit in the laboratory is reported as mass of the chemical adsorbed per unit mass of rock (mg/g), as per the following equation.

$$\text{Adsorption, mg/g} = \frac{[\text{Initial Conc} - \text{Final Conc.}]\,(\text{mg/L}) \times \text{Vol.of Solution (L)}}{Dry\ weight\ of\ rock\ (g)}$$

where Conc is concentration, mg/L, and Vol is volume, L

Results and discussion

Shale properties and characterization

The cores were imaged using CT scan to enable evaluation of core state (heterogeneity, nodules, fractured, etc.)

and selected for the soaking test. Figure 13 lists the selected sample based on the CT scan image.

XRD results were used to determine the total clay content of samples. Clays information is a necessary prerequisite for assessing the effectiveness of surfactant, especially in investigating the adsorption of surfactants (Amirianshoja et al. 2013). Figure 14 shows the XRD results, where the samples comprise primarily of quartz (38.6%—58.6%), plagioclase (13.4%—20.7%), k-feldspar (0%—11.9%), calcite (6.9%—12.2%), dolomite (6.3%—16.6%), and total clay (1.3% -8.37%). The total clay content comprises of illites (96.4%—98.8%), kaolinite (0.1%—1.6%), Chloride (0—1.7%), mixed layer (0.5%—0.9%), and smectite (0.3%—0.6%). The measured ranges were broad and include different formation classes and depths. As reported by Amirianshoja et al. (2013), the anionic surfactant was less adsorbed and normally negligible in most cases comparing with the non-ionic surfactant. The rank order of adsorption for non-ionic surfactant is illite > kaolinite (Amirianshoja et al. 2013). The adsorption due to kaolinite might be negligible since the total clay content is small.

Javaheri et al. (2018) investigated the plugs cored from a well in the Gordondale area, which only covers the upper and lower Montney formations with a depth of 2100–2200 m. The XRD results showed the samples are made up of quartz (40%-45%), k-feldspar (9%-15%), plagioclase (10%-16%), dolomite (14%-22%), and clay (9%-14%) (Javaheri et al. 2018).
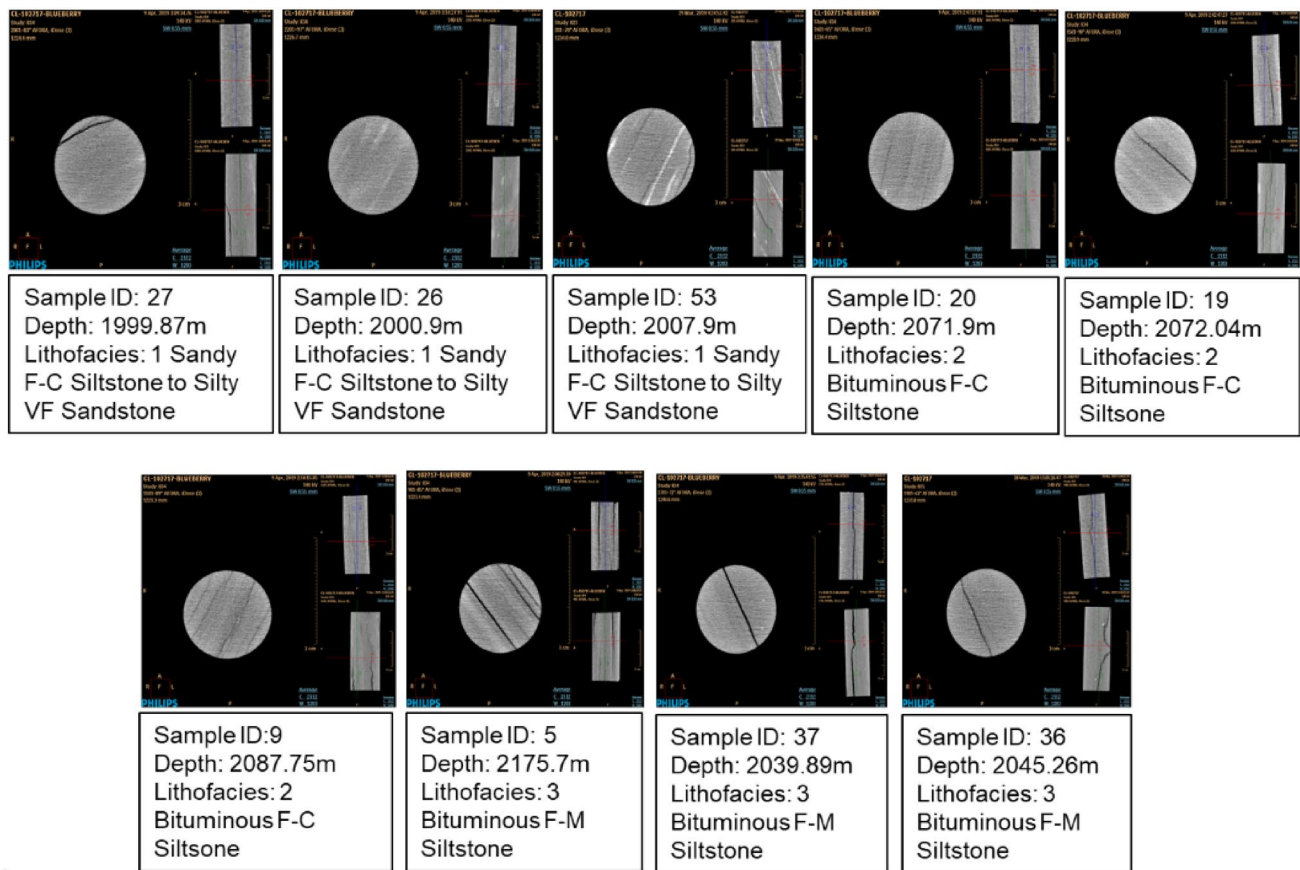
Fig. 13 CT Scan Images of Selected Core Samples for Surfactant Performance Fluid-rock Interaction
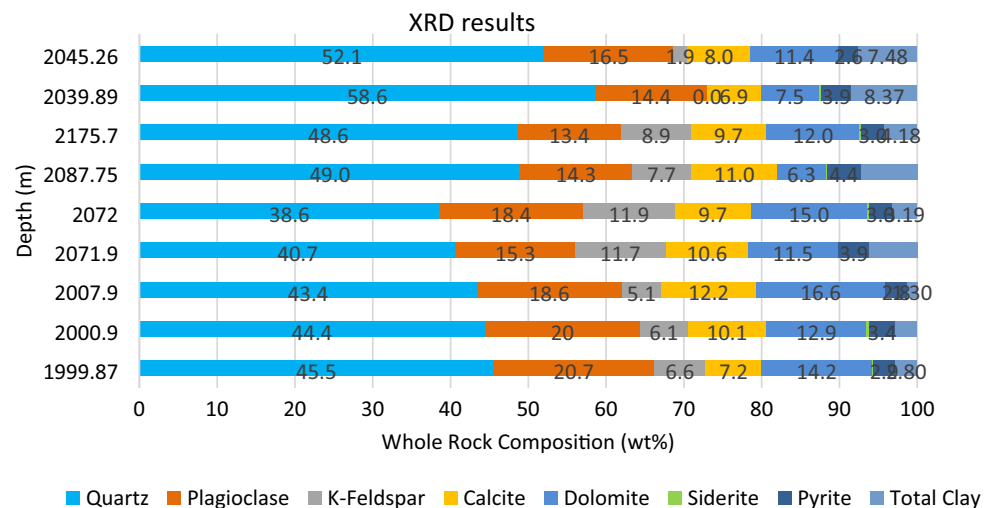
Fig. 14 XRD Results

Figure 15 shows the capillary pressure and pore throat size distribution. Based on the capillary pressure curve and pore size distribution, lithofacies 3 is tighter than lithofacies 1 and 2. The pore size distribution for all lithofacies showed that the Montney shale samples have micropore throat radius.
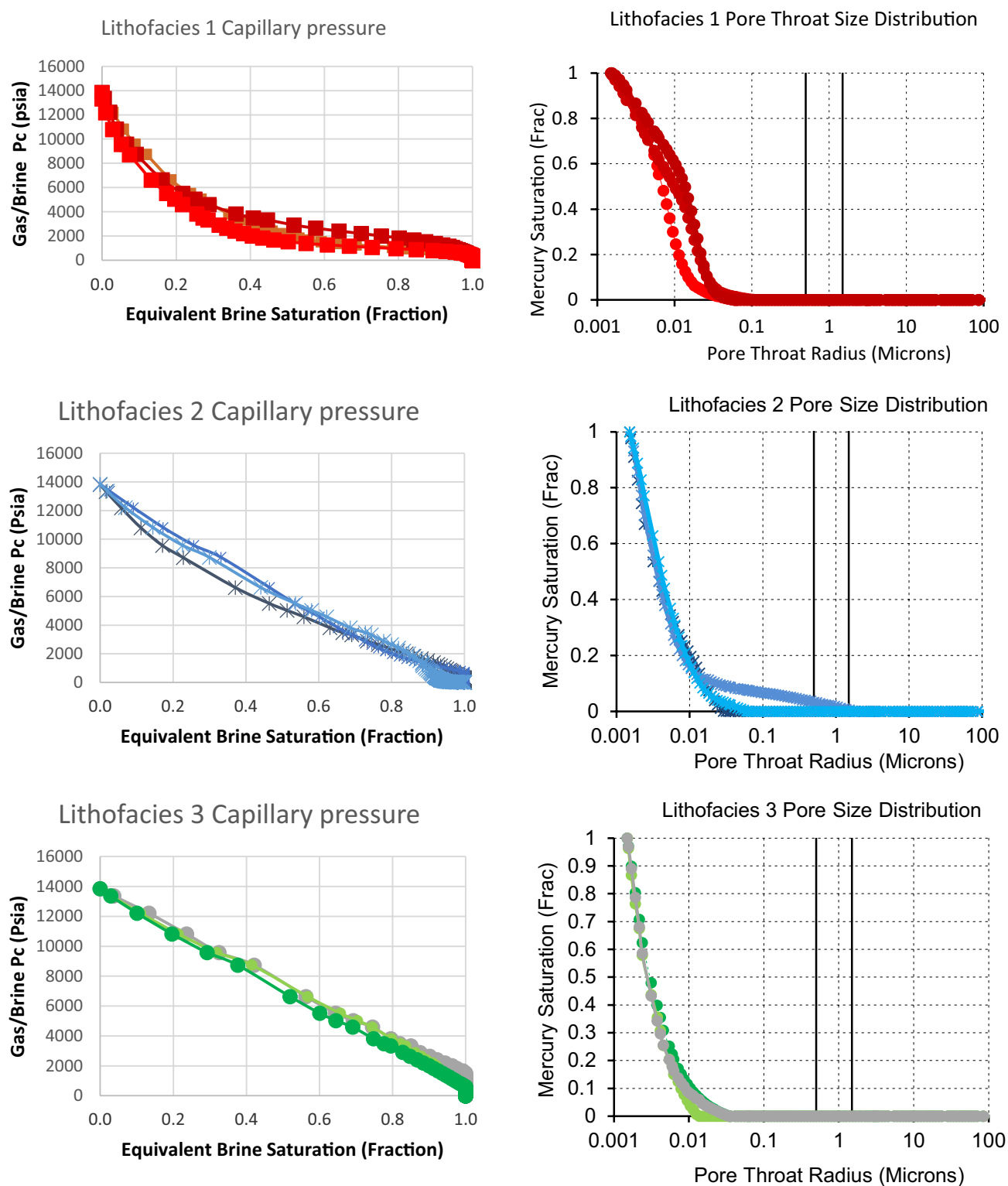
Fig. 15 Capillary Pressure and Pore Size Distribution

Surfactant screening

The sample of 22 surfactants identified for screening was sourced from chemical vendor suppliers. Figure 16 presents the results for the screening. Three of the surfactants, Glu-coPure Wet, SOLOTERRA 938, and Aspiro S2420X, meet the IFT key performance index of less than 0.1 mN/m and surface tension key performance of less than 28 mN/m.
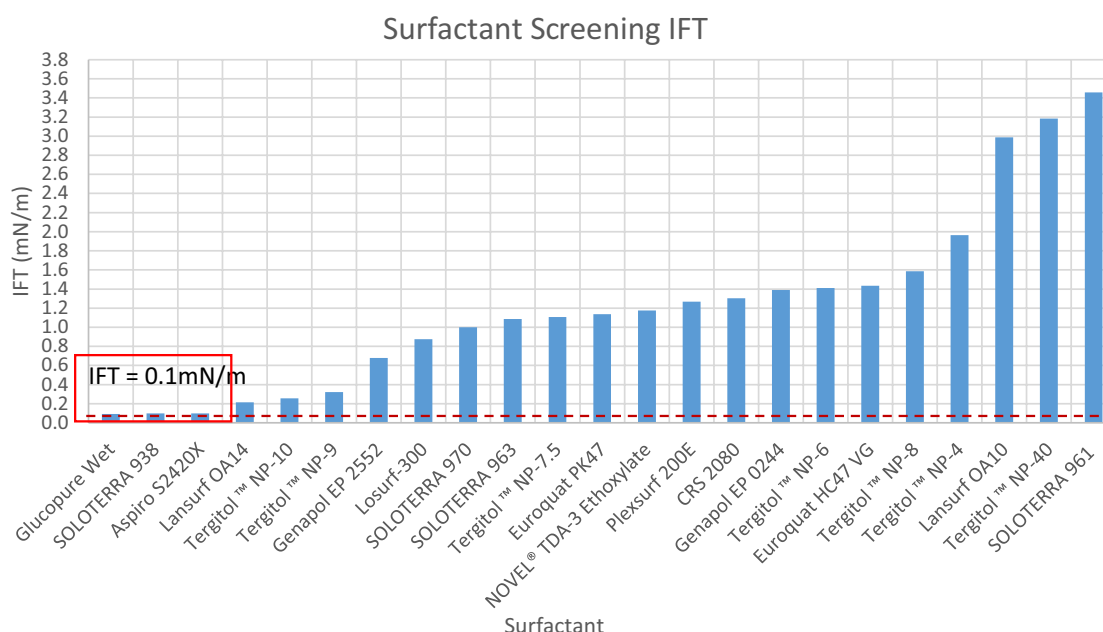
Fig. 16 IFT measurement of 22 Screened Surfactants
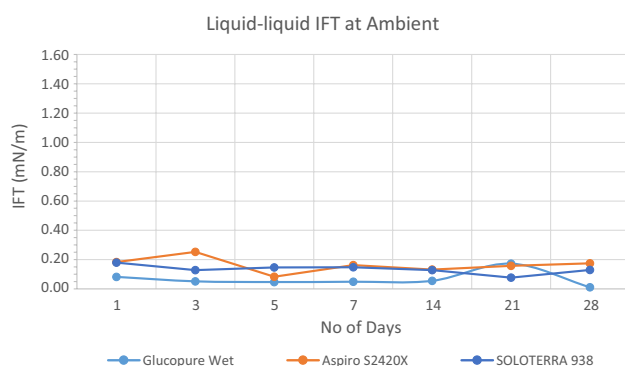
Fig. 17 Liquid–liquid IFT for Shortlisted Alternative Surfactants
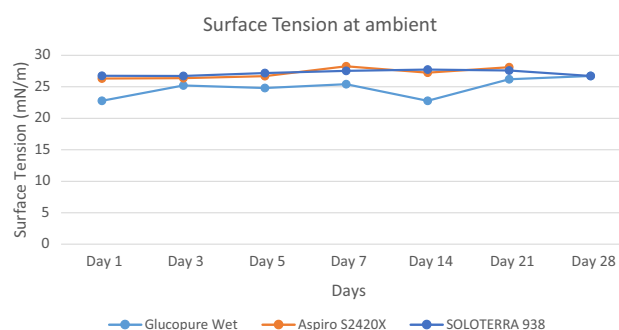
Fig. 18 Surface Tension for Shortlisted Alternative Surfactants

IFT and surface tension key performance was based on the trends identified from a chemical company survey. The survey results showed the IFT between surfactant and Montney flowback water screening ranges between 0.09 mN/m and 3.46 mN/m.

Interfacial tension and surface tension

The 28-day liquid–liquid IFT and surface tension for SOLOTERRA 938, Aspiro S2420X, and GlucoPure Wet were measured. The analysis was conducted for 28 day based on the field implementation duration where fluid–fluid interaction is ranging from 7 to 28 day. Figure 17 and Fig. 18 show that the IFT and surface tension for all surfactants remain unchanged throughout the 28-day stability test at reservoir

temperature. The 28-day stability test was carried out to determine and eliminate the surfactant degradation factor caused by elevated temperature and interaction duration since surfactant degradation affects its performance (SalahEldin Hussien et al. 2019).

The effect of soaking test with reservoir rock on pH measurement

The soaking test was performed on GlucoPure Wet, Aspiro S2420X, and SOLOTERRA 938. The fluids were tested for changes in pH, surface tension, interfacial tension, concentration, and adsorption before and after soaking.

SOLOTERRA 938 (ethoxylated carboxylate) is an anionic surfactant but exhibits non-ionic surfactant properties in an acidic environment. SOLOTERRA 938 is neutralized

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with alkali to form ether carboxylates. According to the Technical Data Sheet (TDS), this surfactant is acidic by nature and has a pH value of 3. Internal laboratory measurement showed that SOLOTERRA 938 has a pH of 3.17. Figure 19 shows the change in the pH values before and after soaking. The pH for the mixed water is 6.45, and when SOLOTERRA 938 is diluted in the mixed water, its pH increased slightly to 3.81 for the pre-soaked fluid. The pH for lithofacies 1, 2, and 3 increased from 3.81 to 6.37 after seven days of fluid interaction with reservoir rock. In summary, SOLOTERRA 938 behaved as a non-ionic surfactant in the pre-soaked fluid, and its behavior changed to anionic SOLOTERRA 938 after seven days of interaction with the reservoir rock. GlucoPure Wet and Aspiro S2420X did not show significant changes after seven days of interaction with the reservoir rock. This behavior is similar to base water without surfactant.

pH measurement is critical for pH-sensitive surfactant because surfactant performance varies with pH. SOLO-TERRA 938, GlucoPure Wet, and Aspiro S2420X are pH-sensitive surfactants, where the measured surface tension of the surfactants varies with pH value. A solution pH higher than surface point (zero of charge) is preferred to reduce surfactants adsorption (Zulkifli et al. 2020). Figure 19 shows that the pH for GlucoPure Wet and Aspiro S2420X is stable, but the pH for SOLOTERRA 938 increased from acidic to neutral after seven days of fluid-rock interaction.

The effect of soaking test on surface tension

The surface tension of pre-soaking and post-soaking fluids interacting with the rock was measured using the Pendant Drop tensiometer. Results showed that the surface tension for GlucoPure Wet and Aspiro S2420X increased after seven days of fluid-rock interaction at reservoir pressure and temperature. However, the surface tension for SOLOTERRA 938 decreased slightly after seven days of soaking. Figure 20 and Fig. 21 show that this occurred for both air and hydrocarbon gas.

Table 5 presents the result of SOLOTERRA 938, which is similar to that reported by Chiappisi (2017). The surface tension for polyoxyethylene alkyl ether carboxylic acids decreased with lower concentrations at different pH values and increased after a specific concentration. In this study, the value for surface tension is influenced by pH value due to the pH-sensitive surfactant behavior. The surfactant performance varies with pH value.

The effect of soaking test on IFT

Figure 22 presents the IFT measurement, which shows that SOLOTERRA 938 retained an IFT of 0.1 mN/m after seven days of soaking. This indicates that it has a good performance with low interfacial tension at reservoir condition for seven days. Aspiro S2420X showed a minimal increase on IFT from 0.1 mN/m to 0.14 mN/m after the soaking test. Aspiro S2420X recorded a low IFT value at a low concentration at the spinning drop tubing. The concentration test using HPLC confirmed that the surfactant was adsorbed on the rock surface. The surfactant adsorption was evident by the reduced surfactant concentration and higher IFT value and surface tension, all of which reduced surfactant efficiency (Belhaj et al. 2020). However, GlucoPure Wet showed a significant loss of performance. The increase in IFT from

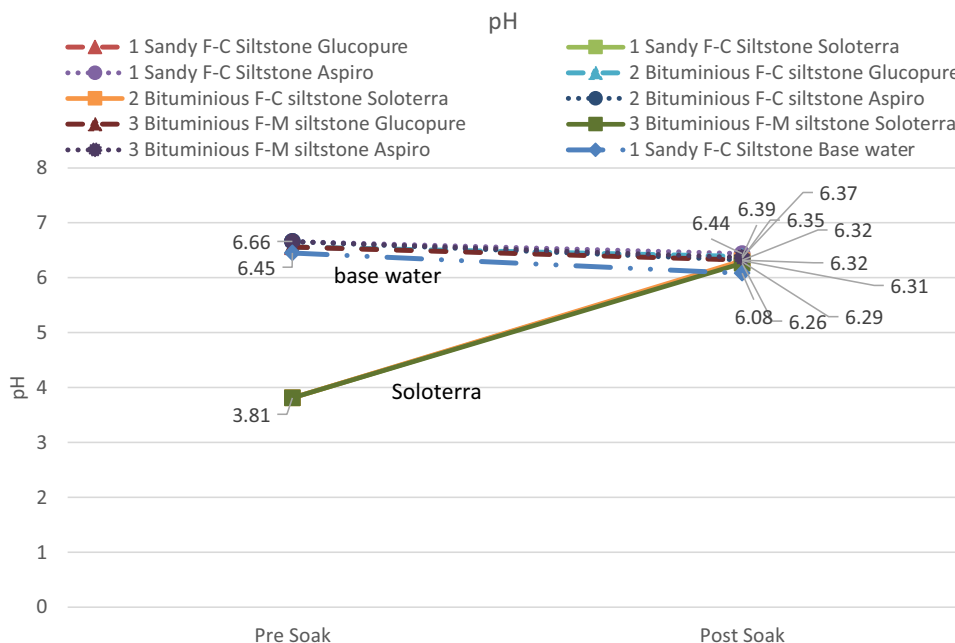**Fig. 19** Comparison for Pre- and Post-soaking Fluids

Fig. 20 Pendant Drop Surface Tension with Air at Ambient Condition for Pre- and Post-soaking Fluids

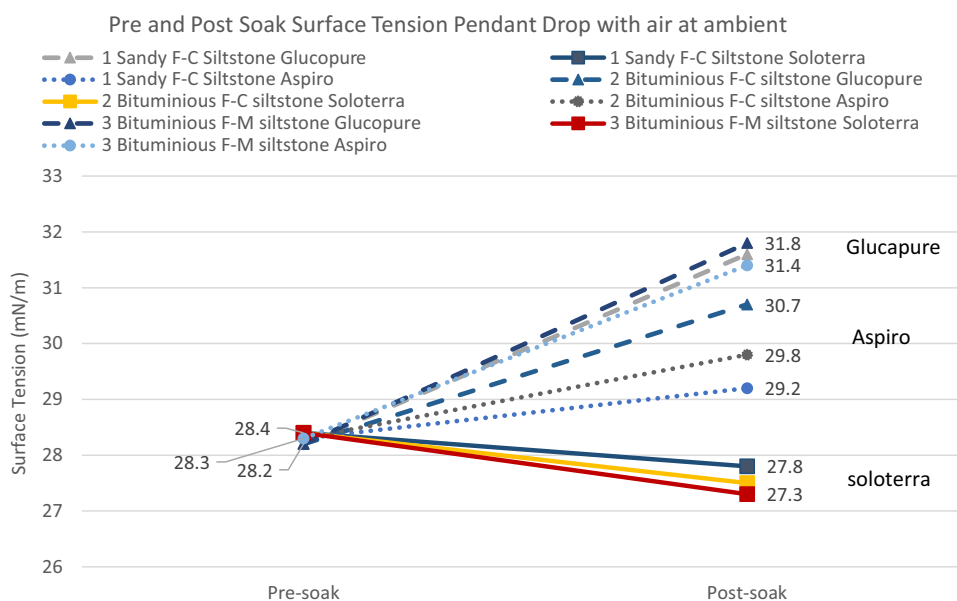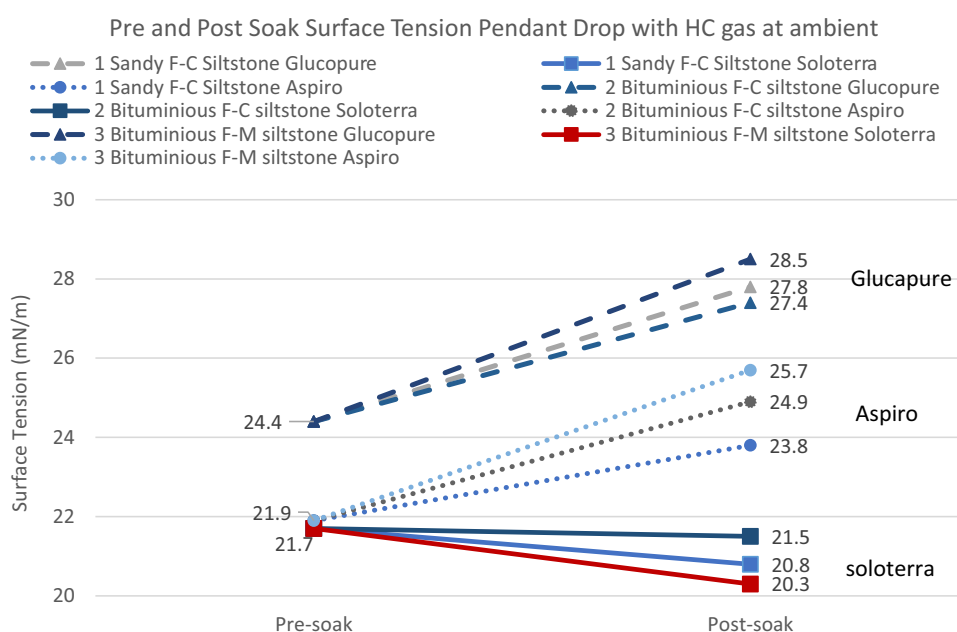Pre and Post Soak Surface Tension Pendant Drop with air at ambient

Fig. 21 Pendant Drop Surface Tension with Hydrocarbon Gas at Ambient Condition for Pre- and Post-soaking Fluids

Pre and Post Soak Surface Tension Pendant Drop with HC gas at ambient

Table 5 Summary of Pre- and Post-soaking Fluids Tests of SOLO-TERRA 938

Parameter	Pre-soaking	Post-soaking
Concentration (wt %)	0.1	0.088
pH	3.81	6.31
Surface tension with air (mN/m)	28.4	27.5
Surface tension with hydrocarbon gas (mN/m)	21.7	20.8

0.003 to 0.15 mN/m and the loss of 34% to 38.8% surfactant concentration indicate surfactant adsorption.

The effect of soaking test on concentration and adsorption measurement

High-performance liquid chromatography (HPLC) was used to measure the active concentration in the pre-soaking and

Fig. 22 IFT for Pre- and Post-soaking Test Fluids

Pre and Post Soaking Test (Liquid-liquid IFT)
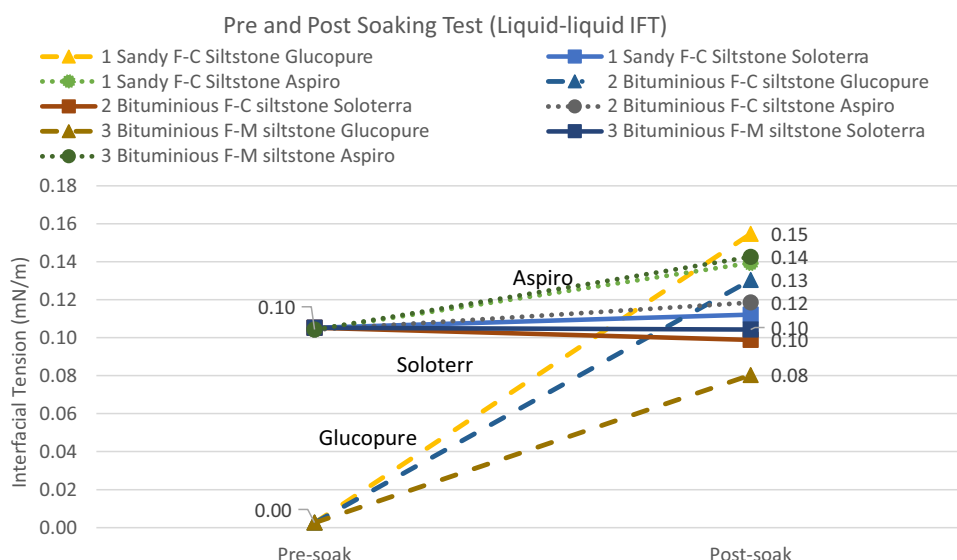
Fig. 23 Concentration Comparison of Pre- and Post-soaking Test Fluids

HPLC Concentration on Pre and Post Soaking test Fluids
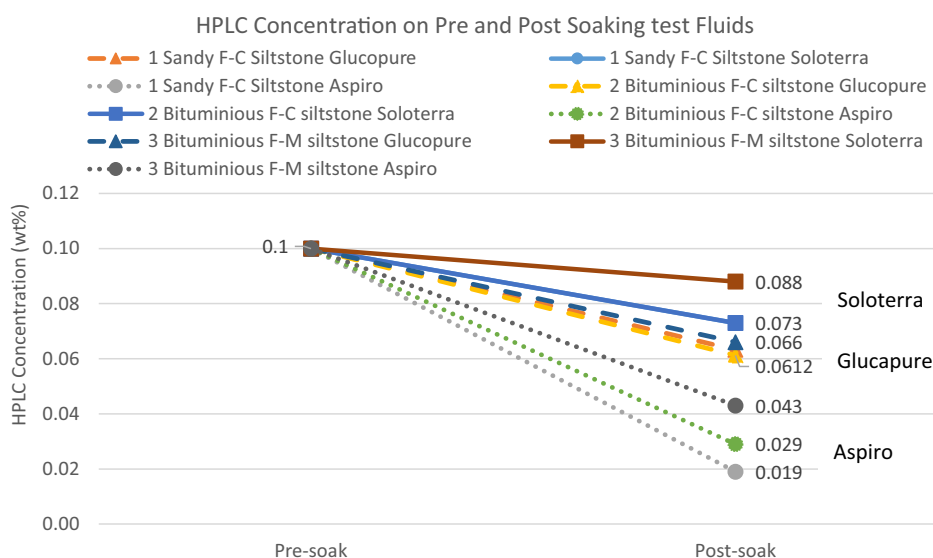
post-soaking test solution. The concentration was used as an input in surfactant adsorption calculation. The general understanding is that the adsorption of charged surfactant onto a surface is due to the cation exchange mechanism.

Figure 23 and Fig. 24 show the changes in concentration and adsorption. Aspiro S2420X showed the highest concentration loss after seven days of soaking with the reservoir rock. The concentration loss ranged from 57 to 81% and contributed to the high adsorption that ranges from 2.043 to 2.903 mg/g rock. This type of surfactant is not suitable for the Montney shale formation. The pH value affects the adsorption level. GlucoPure Wet has a low concentration loss of between 34% and 38.8%, which results in low adsorption of 1.219 to 1.391 mg/g rock. However, the increasing IFT and surface tension values make this type of surfactant

unsuitable for Montney shale formation. The loss of SOLO-TERRA 938 concentration is minimal and ranges between 12 and 27%, while its adsorption value ranges between 0.43 and 0.968 mg/g rock. Based on the data, SOLOTERRA 938 shows good surfactant performance efficiency for Montney shale formation and condition.

pH is one of the key factors affecting adsorption, especially for pH-sensitive surfactants since surfactant performance varies with pH value. SOLOTERRA 938, GlucoPure Wet, and Aspiro S2420X are pH-sensitive surfactants. The pH of GlucoPure Wet and Aspiro S2420X did not vary for pre-soaking and post-soaking test fluids. The adsorption performance of these surfactants is not influenced by pH but is due to cation exchange.

Fig. 24 Adsorption Comparison of Pre- and Post-soaking Test Fluids
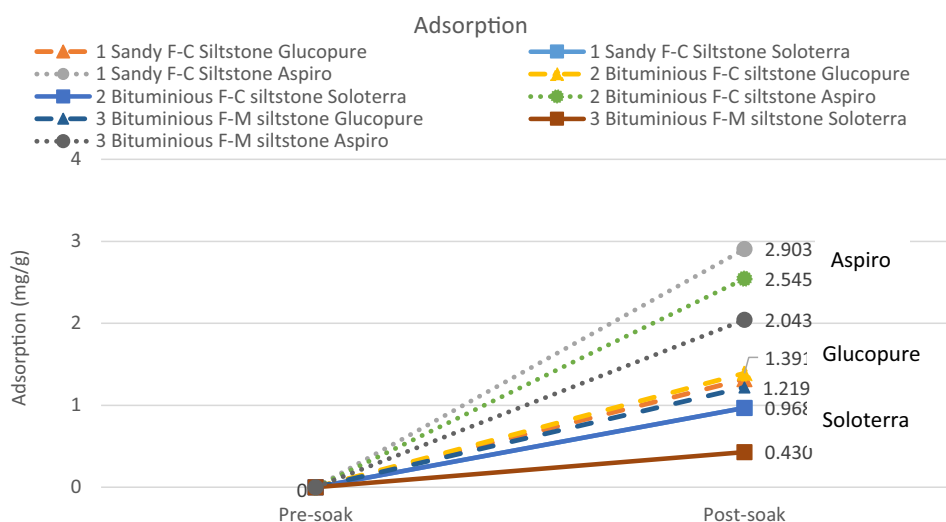
The pH for SOLOTERRA 938 increased from 3.81 to 6.37. SOLOTERRA 938 is an anionic surfactant although it has the typical properties of a non-ionic surfactant in an acidic environment. SOLOTERRA 938 behaved as a non-ionic surfactant during the pre-soaking test and changed its behavior to anionic surfactant after seven days of interaction with reservoir rock. The adsorption occurred during the early stage of soaking due to the non-ionic behavior of SOLOTERRA 938 at a pH of 3.81, where it was adsorbed on the rock surface through the cation exchange mechanism. Acetic acid has the same nature as SOLOTERRA 938 and may have free ions that attack the rock surface charges due to the dipole interaction and cause the fluid to be neutralized during the seven-day soaking period. This mechanism does not affect surfactant adsorption and concentration. Chiappisi (2017) recorded a lower activity coefficient of surfactant, which resulted in minimum surface tension at high pH, as observed in this study.

Conclusion

An assessment of 22 surfactants showed that ethoxylated carboxylate is the suitable surfactant for Montney shale formation because

- The IFT remained 0.1 mN/m after seven days of interaction with the reservoir rock.
- The surface tension remained unchanged after seven days of interaction with the reservoir rock with both air and hydrocarbon gas at ambient temperature.
- The loss of surfactant concentration is 12% after seven days of interaction with the reservoir rock (from 0.1 to 0.088wt%).

- The adsorption value is low and ranges between 0.43 and 0.968 mg/g rock.

Declarations

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