

**CHARACTERISTICS AND REMOVAL OF FILTER CAKE FORMED BY  
FORMATE-BASED DRILLING MUD**

A Thesis

by

MOHAMMED B. ALOTAIBI

Submitted to the Office of Graduate Studies of  
Texas A&M University  
in partial fulfillment of the requirements for the degree of  
MASTER OF SCIENCE

May 2008

Major Subject: Petroleum Engineering

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Approved by:

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## ABSTRACT

Characteristics and Removal of Filter Cake Formed by  
Formate-Based Drilling Mud. (May 2008)

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Formate-based mud has been used to drill deep gas wells in Saudi Arabia since 2004. This mud typically contains XC-polymer, starch, polyanionic cellulose, and a relatively small amount of calcium carbonate particles, and is used to drill a deep sandstone reservoir (310°F). Calcium carbonate particles are frequently used as weighting material to maintain the pressure that is required for well control and minimize the leak-off. Such solids become consolidated and trapped in the polymeric material and this makes the filter cake a strong permeability barrier.

Various cleaning fluids were proposed to remove drilling mud filter cake; including: solid-free formate brine and formate brine doped with organic acids (acetic, formic, and citric acids), esters, and enzymes. The main objective of this research is to assess the effectiveness of these cleaning fluids in removing drilling mud filter cake. A dynamic high-pressure/high-temperature (HPHT) cell was used to determine characteristics of the drilling mud filter cake. Drilling mud and completion fluids were obtained from the field. Compatibility tests between potassium formate brine, cleaning

fluids, and formation brine were performed at 300°F and 200 psi using HPHT visual cells. Surface tensions of various cleaning fluids were also measured at high temperatures.

The conventional method for cleaning the filter cake is by circulating solid-free formate brines at a high flow rate. This mechanical technique removes only the external drilling fluid damage. Citric acid at 10 wt%, formic acid, and lactic acid were found to be incompatible with formate brine at room temperature. However, these acids were compatible with formate brine at temperatures greater than 122°F. Only acetic acid was compatible with formate brine. A formula was developed that is compatible at room and reservoir temperature. This formula was effective in removing filter cake. A corrosion inhibitor was added to protect downhole tubulars. In general detail, this research will discuss the development of this formula and all tests that led to its development.

## **DEDICATION**

This work is dedicated to:  
my lovely supportive parents,  
my beloved patient wife, Taiba  
and my wonderful children, Muhand and Raghad  
with love and appreciation.

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All kinds of praise and all thanks belong to the **God**, the **One**, the **Lord** of the **Universe**, the **Creator**, the **Most Gracious** and the **Most Merciful**. Deep affection is especially due to my parents who supported and encouraged me throughout my study. My love and thanks to my wife for her patience and support while I was studying for my degree.

I would like to thank Saudi Aramco for giving me this opportunity to further my education to receive a Master of Science Degree.

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## CHAPTER I

### INTRODUCTION

The objective of multilateral-well technology is to improve the well productivity by maximizing reservoir contact, resulting in field development with a fewer number of wells. Long horizontal wells (up to eight km) are drilled in Saudi Arabia fields, but the greatest opportunity, and technological challenge, lies with MRC wells. A Maximum Reservoir Contact (MRC) well definition is a long horizontal well with more than five km of total contact with the reservoir rock (Hussain et al., 2005).

It is well recognized that near-wellbore flow properties are altered by drilling-fluid and fluid filtrate invasion during overbalanced drilling operations. The degree of alteration, generally called formation damage, depends on a large number of parameters, such as the nature and characteristics of the drilling fluid, formation properties, and operating conditions (Ding et al., 2006).

Formation damage caused by drilling-fluid invasion may create substantial reductions in oil and gas production rate in many wells. Productivity losses are especially critical for long horizontal wells, which are often have an openhole completion. In such cases, the near-wellbore damage is not bypassed by perforations and may lead to very large skin values. Therefore, prevention of formation damage generated by a drilling fluid may not always be possible because, first, the drilling time of the horizontal segment in the producing zone is usually many times longer than in a typical

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This thesis follows the style of *SPE Journal*.

vertical well, leading to a much deeper filtrate invasion; and second, the very low drawdown pressure that is needed to produce from a typical horizontal well reduces the viscous forces available to cleanup near-wellbore damage (Yildiz, 2006; Longeron et al., 2000).

Formation damage and the subsequent increase in skin factor was influenced primarily by the type and properties of the drilling fluid used in the reservoir section, but can be limited by careful attention to drilling practices while in the reservoir. Skin can result from unnecessary and repeated damage to the mud filter cake during drilling, tripping, and reaming operations (Liu and Civan, 1994).

### **1.1. Drilling Fluids Background**

Drilling fluids are either water-based or oil-based. These fluids are made up of a base fluid, water or diesel (HC), weighting agents and other additives that aid in removing the cuttings from the well and keep the mud in a fluid state. Traditionally, the drilling industry has used water-based muds (WBMs) because they are inexpensive and have minimal impact on the surrounding environment. The density of the WBMs can be increased by adding calcium carbonate particles. High levels of suspended solids, however, in drilling fluids can cause high frictional losses during fluid circulation. These losses create high ECDs and limit pump rates. Low pump rates can result in inefficient hole cleaning (Njobuenwu and Wobo, 2007).

In difficult drilling situations, such as horizontal, extended reach, deep wells, and reactive shales, drillers rely usually on oil-based muds (OBMs). However, despite their



high-performance properties, OBMs are costly to dispose-off and contain toxic materials, such as mineral oil.

The main concern with WBM is the thermal degradation of chemical additives that often occurs while drilling high temperature wells. Such degradation can lead to strong variations in rheological and filtration characteristics and loss of fluid properties (Melbouci and Sau Arjun, 2006). Moreover, even if no degradation of components occurs, the viscosity of hydro-soluble polymer solutions commonly used in drilling fluid formulations strongly decreases at high temperatures. Improperly formulated and maintained drilling fluid systems can cause significant near-wellbore formation damage and create potential for the plugging of screens and slotted liners (Pitoni et al., 1999).

The usual problems that encountered with conventional KCl/polymers drilling fluid in gas wells in Saudi Arabia are:

- Downhole fluid and barite losses in the horizontal section. The leak-off also happens during the completion phase when drilling fluid lose their suspension properties at high bottom hole temperatures due to degradation of the polymers (Saasen et al., 2002; Bradshaw et al., 2006).
- Differential sticking due to large pressure overbalance (800-1,600 psi). Sometimes, sloughing shales, which interbedding the main gas reservoirs, need to be stabilized (Simpson et al., 2005).
- Excessively high rheological properties, resulting in unacceptable surface pump pressures when conventional non-damaging brines such as calcium chloride and

calcium bromide are used as base fluids with calcium carbonate as a weighing and bridging agent (Oswald et al., 2006).

Using formate based mud can solve these concerns that have been usually encountered with conventional water and oil-based muds (Svendsen et al., 1998; Svoboda, 2002; Messler et al., 2004; Downs et al., 2006).

## **1.2. Solids-free Formate Brines**

Solids-free formate-based brines were first recognized as having the ability to extend the thermal stability of bio-polymers in the late 1980s and early 1990s. Their first applications in the field were in the early 1990s, and their use as the basis of reservoir-drilling and completion fluids has become widespread since then (Downs, 1993). Their ability to preserve conventional polymers at temperatures greater than 250°F, and in some cases up to 360°F, has been the primary reason for their selection (Messler et al., 2004). Polymer stabilization in a formate brine is typically achieved by the promotion of hydrogen bonding. This promotion raises the transition temperature and thus maintaining the fluid viscosity at elevated temperatures (Lomba et al., 2002). The formate salts also have an unusually high solubility in water, at a wide range of densities, and reduce the rate of hydrolytic and oxidative degradation of many viscosifiers and fluid loss agents at high temperatures (Downs et al., 2005). Formate also has the advantage that much of the weight is provided by the base brine resulting in lower solids content (Weekse et al., 2002). Sodium and potassium formate based drilling fluid have been used in drilling HPHT gas wells in Saudi Arabia since 2004 (Simpson et al., 2005).

Formate brine also has the ability to delay cross-linking time for gel fluids in conformance control operation. It has been used due to the high density that can be sustained with this type of brines. At higher densities, formate brines tend to precipitate out the polymers and cross-linkers (Bouwmeester and Van Gijtenbeek, 2005).

### **1.3. Compatibility of Formate Brines with Other Fluids**

Compatibility of formate-based drilling fluid with formation brine was discussed in several publications (Downs et al., 2005; Hands et al., 1998; Byrne et al., 2002). Byrne et al. presented specific procedure for formate testing at reservoir conditions. The conclusion from Byrne paper was that the formate-based fluid is compatible with formation brines at high temperatures and the common precipitation was noticed only at room temperature condition. Acetic and formic acids at a concentration of 15 wt% were prepared in a formate brine solution to remove the filter cake damage. The compatibility of the organic acids with formate brine was not discussed although they were used in that paper. There was no evidence of adverse formate/formation brine reaction at reservoir conditions. Some of the formation brines incorporated in these tests were highly saline with salt saturations greater than 200,000 ppm.

Downs et al. proved that there might be precipitate from mixing formate brine solution with the reservoir fluids (Downs et al., 2005). They concluded that any precipitates formed are not scale-i.e. they are inherently water-soluble (e.g. KCl). The precipitates that created in the lab experiments might be due to the unrealistic mixing ratios and room temperature condition.

A recent study on formate brine compatibility with formation brine was published in last April (Javora et al., 2007). Several tests were conducted at three ratios (75/25, 50/50, and 25/75) and at temperatures 70 and 180°F. They noticed some solids precipitates and the amount of those solids depended on two conditions: temperature and mixing ratio. The composition of the solids was not identified in Javora et al. paper.

Svendsen et al. tested citric and acetic acids at a concentration of 10 wt% to remove filter cake damage that generated by formate based mud (Svendsen et al., 1995). They found the citric acid was better than acetic acid. The organic acids compatibility with formate brine solution was not studied in that paper.

Larsson and Nahringbauer presented a procedure to prepare a potassium hydrogen diformate in the lab by dissolving recrystallized potassium formate in formic acid during heating (Larsson and Nahringbauer, 1968). The salt crystals were found to be unsuitable for X-ray work. XRD and SEM analysis result in my research study shows the potassium hydrogen diformate was present when formic acid mixed with formate brine. Potassium hydrogen citrate, also, was noticed when citric acid was mixed with formate brine. More details about potassium hydrogen diformae and citrate will be shown in Chapter IV.

#### **1.4. Conventional Filter Cake Clean-up Techniques**

The clean-up of the formate drill-in fluid (DIF) filter cake that deposited in long horizontal sections is a permanent problem. Traditionally, cleaning up a reservoir filter cake has been accomplished with conventional chemical and mechanical means. The chemical means include formate brines (Hands et al., 1998), live mineral and organic

acids (Huang et al., 2000), enzymes (McKay et al., 2000; Al Otaibi and Nasr-El-Din, 2005), chelating agents (Bradshaw et al., 2006), or a combination of formate brines and organic acids (Svendsen et al., 1995). The combination of high reservoir temperature and long shut-in times after acid treatment lead to a high probability of severe corrosion of the well completion. In addition to the corrosion concerns with common acids, the rapid removal of the filter cake with strong acids could create localized, high leak-off of the treating fluid resulting in an uneven distribution of acid across the horizontal open-hole section.

Different breakers were used to remove filter cake damage caused by formate-based DIF. The breakers were composed mainly of potassium formate mixed with different organic acids, including: 10 wt% citric acid and a dispersant, 10 wt% acetic acid, 10 wt% citric acid, and a proprietary breaker formulation. The highest removal efficiency was 90%, which was attained by potassium formate brine mixed with 10% citric acid. The other three fluids gave insufficient removing efficiency, which was below 70% (Svendsen et al., 1995).

Hands et al. compared the clean-up issues of oil-based mud and formate brine system. Mechanical (jetting) and chemical (acids) means were tested in the lab to assist with drilling fluid selection and mud cake clean-up. Another mud cake clean-up test was also mentioned by using drawdown pressure alone. In general, jetting techniques did not contribute much to the clean-up process compared to the drawdown test. The formate system gave better return permeability after drawdown than oil based mud. Acid and acid/solvent mixtures had been used to remove the mud filter cake for the two drilling

fluid systems. Return permeability results were high for the formate brine systems compared to the oil based mud (Hands et al., 1999).

Hands et al. also utilized the sodium/potassium formate brines to remove the filter cake formed by sodium formate DIF, which included some calcium carbonate particles, xanthan and starch polymers. The formate brines blend (sodium/potassium) was used, without any solids additives, in a sandstone gas well. The well production rate was high and for that reason no acid stimulation job was recommended (Hands et al., 1998).

More than 40 core flood tests were conducted by Byrne et al. The conclusion from those tests was recommending using the formate DIF since it gave better return permeability results compared to conventional oil or water-based mud. Filter cake damage, by formate DIF, was cleaned by using 15 wt% acetic acid mixed with solids-free formate brine. 15 wt% formic acid also was used, however it did not cause any matrix dissolution compared to HCl (Byrne et al., 2002).

The formate-based reservoir drilling fluid can be processed through normal solids-control equipment to remove the majority of bridging agent and drill solids. The processed fluid can be reused as a completion fluid during the completion phase, thus saving the additional cost from the operator for solids-free completion brine (Bungert et al., 2000).

In order to avoid using highly reactive acids, other fluids have been exploited such as enzymes to break down biopolymers in the cake to simple glucose units. McKay et al. reported a case history of clay inhibition in the reservoirs by using a low solids

formate based mud system. Formate blend had been used as a drilling fluid instead of synthetic oil-based mud. A mixture of two enzymes systems was applied in this treatment to remove the filter cake and to enhance the cleanup/back production of filter cake through the oversized gravel. The enzyme breakers were incorporated within the gravel pack carrier fluid (a VES solution) in order to degrade the filter cake polymers (McKay et al., 2000).

Enzymes, however, have some limitations; for example they will only attack specific polymers chains, and the calcium carbonate components will not be removed (Battistel et al., 2005). Enzymes are also quickly denatured and lose their activity and might even represent a potential source of formation damage at high temperatures.

Chelating agent was used by Bradshaw et al. as a breaker to remove filter cake damage (Bradshaw et al., 2006). The sodium formate drilling fluid was displaced by using the same fluid but solids-free. Then, the open hole was displaced to the chelating agent breaker solution. The soaking time, for different wells, ranged from 15 to 110 days.

Oxidizers can be used to degrade the polymers in common water-based drilling fluid filter cakes. The oxidizers work by generating free radicals that attack the links between the polysaccharide components of the polymer. Attack can occur on the polymer backbone or a side chain. Typical polymeric components of water-based drilling fluid filter cakes that can be degraded by oxidizers are xanthan gum, scleroglucan, and starches. The effectiveness of oxidizers is limited by pH (practical pH range is 4 to 10). Oxidizer efficiency is also dependent on temperature (practical range

of most is between 120 and 250°F). Below 130°F, oxidizer effectiveness drops dramatically below pH 8. Most of the common oxidizers, also, are not compatible with formate brines as shown in Table 1 (Schlumberger, 2007).

Complete removal of filter cake in long horizontal wellbore by using conventional breakers might be difficult. Reaction rate is the most effective factor in controlling the distribution of the cleaning fluid in the horizontal leg. Acids as an example will react with calcium carbonate very rapidly at downhole condition. Therefore, rapid consumption of calcium carbonate is likely at the point at which acid is introduced to the wellbore. This can lead to localized clean up, excessive local losses and inability to circulate acid to the rest of the wellbore section (Buijse et al., 2004).

Esters had been introduced lately to solve the fluid distribution and corrosion concern which are usually encountered with others conventional breakers (Moses and Harris, 2004). The action of the ester in the clean-up fluid is delayed, i.e., the ester does not dissociate by hydrolysis to produce organic acid and alcohol until after the clean-up fluid has been placed in the wellbore. The reaction products will be generated as solution temperature increased. The ester reaction kinetics and filter cake clean-up by using two different esters were discussed in previous publications (Al Otaibi et al., 2006; Nasr-El-Din et al., 2005).

Lactic, formic and acetic acids had been tested in detail at high temperatures. Formic and lactic acids showed excellent results, within short soaking time, in removing the damage. Ethyl-lactate ester has been included only in this research study for mainly two reasons. First, the generated lactic acid has strength similar to that of formic acid



and both have dissociation constants that are about 10 times greater than that of acetic acid (Leschi et al., 2006). Secondly, the lactate ester hydrolysis reaction to lactic acid depends on the temperature and also the acid concentration increases gradually with time (Al Moajil et al., 2007). Formate ester generates formic acid immediately and it is independent of the reaction time.

**Table 1:** Compatibilities of the most common oxidizers with formate brines

		Oxidizers					
Brine carrier fluid	Formula	Persulfate	Brenate	Peroxides	Calcium Hypochlorite	Sodium Hypochlorite	Lithium Hypochlorite
Sodium formate	NaCHO <sub>2</sub>	No				LTR <sup>1</sup>	
Potassium formate	KCHO <sub>2</sub>						
Cesium formate	CsCHO <sub>2</sub>						

1. Laboratory testing recommended

### 1.5. Objectives

The main objectives of my thesis are to: (1) Study the compatibility of potassium formate brine with formation brine, organic acids, esters, and other cleaning fluids, (2) Evaluate the ability of different organic acids to remove filter cake damage, (3) Study the removing efficiency of lactic acid precursor, at different concentrations, on filter cake, and (4) Compare the mechanical technique by using potassium formate brine at dynamic condition with other chemical means.

## CHAPTER II

### EXPERIMENTAL STUDIES

#### 2.1. Materials

Field DIF samples were used in all lab experiments. Those samples were obtained from a gas field in Saudi Arabia. The type of the drilling fluid used is a potassium formate-polymer system. The composition and properties of a lab drilling fluid sample that had been recommended for field J are given in Table 2. The properties of the field drilling fluid sample were also measured in the lab as shown in Table 3. Solids-free potassium formate brine was utilized to remove the filter cake mechanically. Acid precursor solutions and organic acids were used to dissolve the calcium carbonate particles present in the filter cake.

**Table 2:** Formulation and properties of formate drilling fluid, lab sample

Material	Quantity (per bbl)	Units	Property	Value	Units
Fresh water	0.277 – 0.299	bbl	Density	80 – 90	Ib/ft <sup>3</sup>
Potassium formate (98 pcf)	0.423 – 0.701	bbl	pH	10.5 – 11	-
Defoamer	0.01	gal	Plastic Viscosity	16 – 22	cp
XC-polymer	0.5 – 1	Ib	Yield Point	18 – 25	Ib/100 ft <sup>2</sup>
PAC-Regular	0.5 – 2	bbl	10 sec. gel	5 – 7	Ib/100 ft <sup>2</sup>
PAC-Low Viscosity	2 – 3	Ib	10 min. gel	6 – 10	Ib/100 ft <sup>2</sup>
Soda Ash	0.5 – 1	Ib	HPHT Filtrate, 300–325°F/500 psi	16 – 18	cm <sup>3</sup> /30 min
Sodium bicarbonate	0.2 – 0.5	Ib	HPHT Filter Cake Thickness	1 – 2	33 <sup>nd</sup> in.
CaCO <sub>3</sub> fine	10	Ib	Total Solids	5 – 8	vol %
CaCO <sub>3</sub> medium	10	Ib	Iron Content	<1.5	mg/l

**Table 3:** Properties of formate-based drilling fluid, field sample

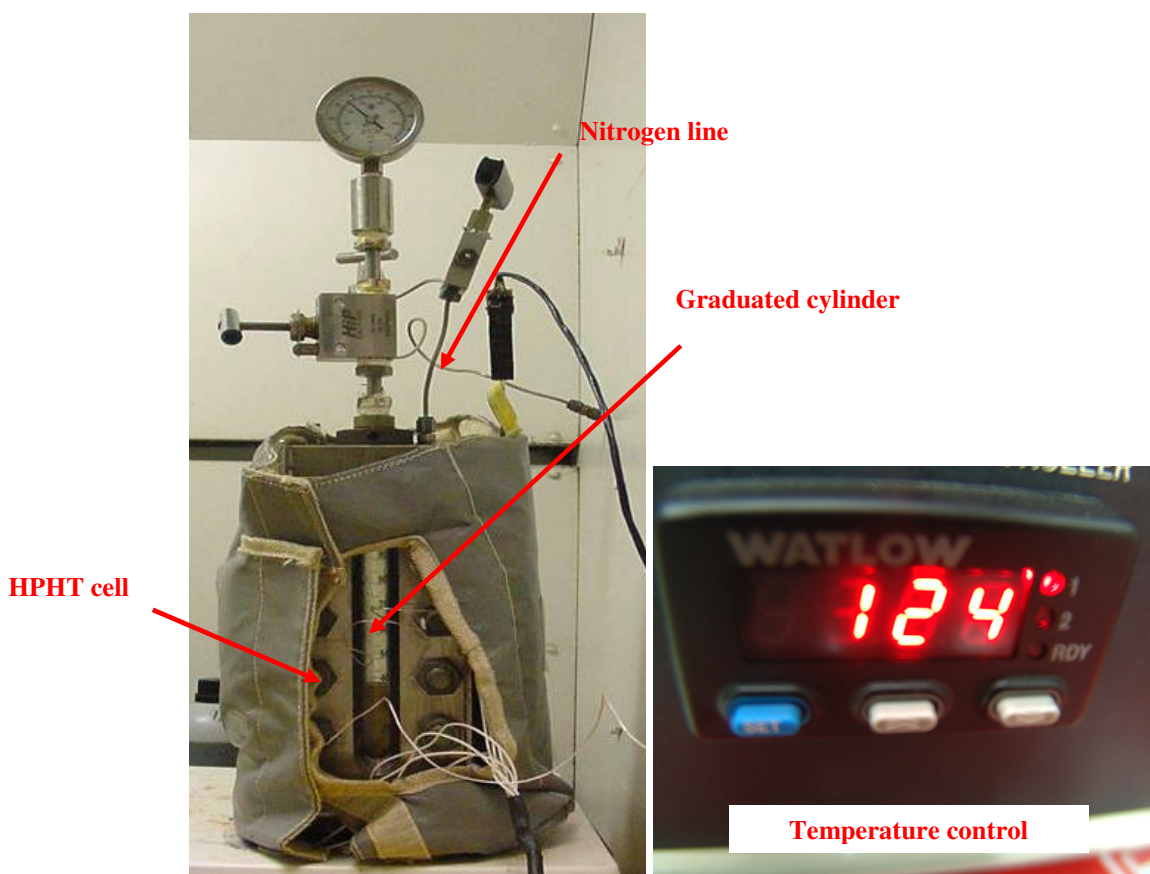
Property	Value	Units
Density	92.6	lb/ft <sup>3</sup>
pH	10.39	-
Plastic Viscosity	17	cp
Yield Point	14	lb/100 ft <sup>2</sup>
10 sec. gel	3	lb/100 ft <sup>2</sup>
10 min. gel	4	lb/100 ft <sup>2</sup>
HPHT Filtrate, 300°F/500 psi	17	cm <sup>3</sup> /30 min
HPHT Filter Cake Thickness	2	33 <sup>rd</sup> in.

## 2.2. Analytical Techniques

Solids present in the filter cake, returned cleaning solution, and mixed fluids were separated, dried and then examined using XRD, XRF and ESEM techniques. The density of various solutions at various temperatures up to 158°F and 14.69 psi was measured using a Paar densitometer model DMA-35. Extrapolation was used in obtaining the cleaning fluids density at higher temperatures. The pH of all solutions examined was measured using an Orion model 250A meter and Cole Parmer Ag/AgCl single junction pH electrode.

## 2.3. HPHT Visual Cell

High pressure high temperature (HPHT) visual cell is used commonly to test the fluids compatibility at reservoir conditions. The loaded sample can be seen through a glass window to observe any chemical reaction or any solids precipitates at high temperatures, Figure 1.



**Figure 1:** HPHT visual cell with automotive temperature control

The steps that are usually followed to load a liquid sample in HPHT visual cell are:

- Use a clean graduated cylinder (100 ml).
- Load the sample in the visual cell and connect the pressure line.
- Apply a pressure by using  $N_2$  (usually from 200 to 300 psi) to avoid any liquid vaporization at high temperature.
- Adjust the cell temperature by using an automotive temperature control and usually it heats up quickly.
- Record any observations (reaction, precipitates...etc) during the soaking time.

#### **2.4. Dynamic HPHT Filter Press Cell**

The Dynamic high pressure high temperature (HPHT) filter press measures filtration properties under varying dynamic down hole conditions, Figure 2. A motor driven shaft fitted with propellers turns at varying speeds inside a standard 500 ml HPHT cell. RPM settings from 1 to 1,600 rpm impart laminar or turbulent flow to the fluid inside the cell, and by varying the shaft length, the shear stress may be increased or decreased. The power is driven to the stirring shaft by a timing belt that is easily accessible for quick adjustment and removal. Other features include a variable speed motor controlled through an SCR controller with RPM's indicated on a digital tachometer. The test procedure is exactly the same as that in the conventional static HPHT filtration test. The only difference is the fluid is circulating inside the cell, while filtrate is being collected. Two interchangeable bottom caps for the cell are provided to accommodate quarter inch thick ceramic or sandstone disks, standard screens as well as conventional filter paper, sintered metal or similar thin materials (OFI Testing Equipment, 2007). Detailed experimental procedure will be discussed in the next section.



**Figure 2:** Dynamic HTHP filter press

## **2.5. Experimental Procedure for Dynamic HPHT Filter Press**

1. To prepare for running a test, the cell is removed from the heating jacket. To do this, loosen the belt tensioning screw at the top of the right side of the motor housing. Loosen the two screws holding the motor clamp and support plate. These screws are on the side of the belt guard in the top of the motor housing cover about 7/8 inches from the edge. Open the belt guard and remove the belt. Disconnect the two water line tubes at the top of the shaft. These lines are for circulating water inside the shaft in order to keep the packing gland at a workable temperature level.
2. Lift the cell out of the heating well. Remove the top packing gland cap and shaft by loosening the set screws enough for the cap to slip out of the cell body.
3. Begin pre-heating the heating jacket to the desired temperature by plugging in the power cord and turning the thermostat to about mid-scale. When the heating jacket is turned off, the thermostat is turned fully counter clockwise and the pilot light will be

on. Place a thermometer in the heating well. The pilot light will turn on when the heating well reaches the temperature for which the thermostat is set.

4. Remove the outlet or bottom cell cap. Inspect and replace where necessary all O-Rings on the cell, cell cap and valve stems. Apply "Never Seez" to all O-Rings as additional protection from the temperature. Invert the cell with the outlet end up and place an O-ring in the cell body O-Ring groove. On top of this place the appropriate filter medium.

**Note A:** To prepare ceramic or sandstone discs for filtration, the disc must first be soaked for at least 10 minutes in the base fluid (water or oil, etc.), otherwise the disc will seal up excessively when pressure is initially applied to the cell.

**Note B:** When filter paper is used above 400°F a stainless steel, Dynalloy filter disk should be used instead of paper filters.

5. Re-install the appropriate cell cap. Tighten the set screws and be sure they are fully seated into the cap detents.
6. Tighten (to close) the bottom or outlet valve stem.
7. Set the cell upright with the filter end down, and fill with fluid from the top. Allow at least 2.5 inches of space from the top of the fluid to the O-Ring groove in order to leave room for heat expansion of the fluid and for the shaft displacement of fluid.
8. Install the propeller on the end of the shaft. It fits over the shaft and is locked in place with a set screw. Be sure to seat the set screw on the flat spot at the end of the shaft. Optional shaft extenders adjust the distance from the filter medium and therefore alters the shear rate. The extenders allow for 1/2 inch adjustments in the

distance between the propeller and the filter medium up to 1 1/2 inch. Also a four blade propeller is available as an optional item.

9. After making sure all inlet O-Rings are installed, greased with "Never Seez" and are in good condition, re-install the gland cap on the top of the cell. Above 300°F the O-Rings should be replaced after each test. Fully tighten the set screws in the detents after the proper alignment is determined.
10. Place the cell in the heating jacket which has been preheating. The cell should drop down over the locator pin in the bottom of the heating well. The cell pressure inlet valve stem will be facing away from the motor housing in order to facilitate fastening the pressure manifold to the stem.
11. The inlet and outlet valve stem should be tightly closed. Attach the high pressure line (with 1,500 psi gauge) from the nitrogen manifold to the inlet valve stem. Completely insert the locking pin, and close the bleeder valve on the manifold block. Follow the same procedure with the low pressure line and connect it to the back pressure receiver.
12. Check the T-Screws on the pressure regulators to be sure they are unscrewed (counter-clockwise) and that no pressure is being applied to the system.
13. Open the nitrogen bottle valve and note the bottle pressure as registered on the middle manifold gauge.
14. For filtration tests above 212°F the sample must be tested under pressure.
15. Close the drain valve on the receiver. This valve is also used as the bleeder valve. With a crescent wrench, open the outlet valve stem 1/2 turn, which will pressurize



the receiver. Open the inlet valve stem to apply the same amount of pressure to the fluid inside the cell.

16. Transfer the thermometer from the heating well to the thermometer well in the cell.

The time required to heat the sample to test temperature will vary from 30 minutes to one hour depending on the test temperature, but it should never exceed one hour.

**Caution:** When the closed cell is heating in the jacket, the pressure in the cell will rise rapidly due to the thermal expansion of the sample. This should be carefully monitored during the heating phase.

17. Replace the belt over the sheaves and tighten the belt tension just enough to prevent

slippage. The tightening screw which is attached to the motor support is a two piece screw which allows for locking the screw to the housing at the proper tension. Also tighten the two motor support clamping screws in the top of the motor housing in from of the belt guard. Loosen these and then re-tighten, if adjustments must be made in the belt tension.

18. Attach the water lines to the swivel at the top of the shaft. The inlet line in the center

of the cap should be connected to a cold water source with the 1/8 inch connector, 1/4 inch plastic hose and fittings provided. The drain line from the center of the swivel body can be run into any convenient drain. Experience will determine the amount of water flow necessary for proper cooling. Do not operate the mixer shaft with the O-Rings in the swivel Dry. The water keeps these O-Rings lubricated and also reduces the load on the motor. If no cooling water is to be used, the swivel body can be removed from the shaft by removing the top retaining ring.

19. Set the mixing speed to the desired value between 100 and 1,600 rpm using the speed control knob on the motor control.

## **2.6. HPHT Pendant Drop Instrument**

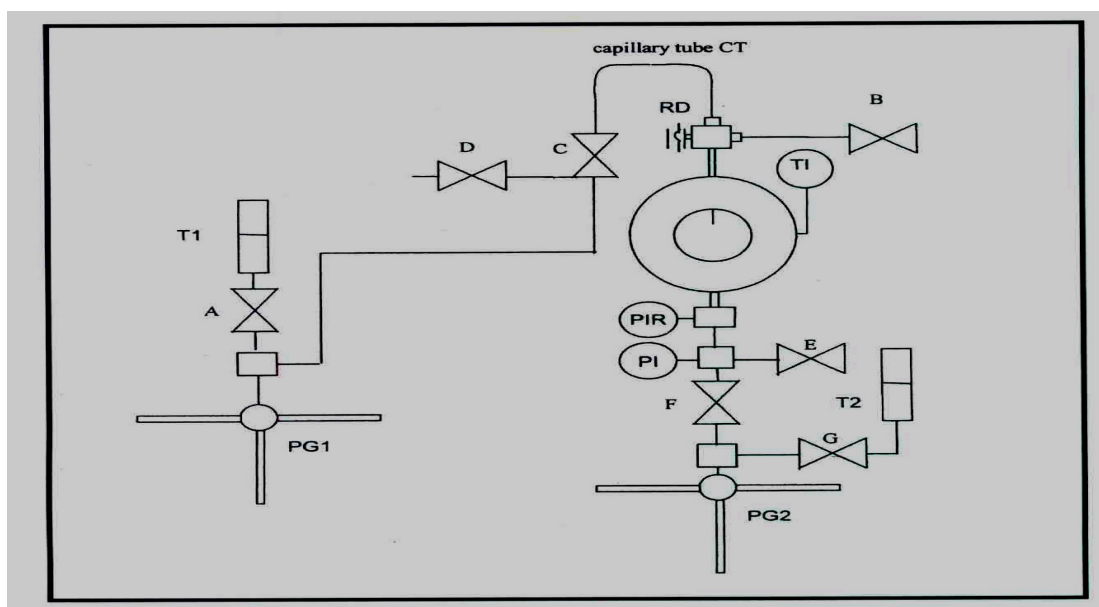
Surface tension of fresh solutions was measured using a high pressure/high temperature pendant drop instrument model PD-E700LL-H manufactured by Eurotechnica Ingenieurbüro GmbH, Germany. The image analysis module of the instrument was a Krüss model DSA10-MK2L. The wetted surfaces of the instrument (the cell and needle) were constructed from Hastelloy-C, which can resist corrosion at high temperatures.

Surface tension measurements were carried out by generating a pendant drop at the end of a capillary tube (OD = 1.575 mm) in compressed nitrogen gas. An image of the drop was recorded using a digital camera (Teli CCD Camera CS8420C-02) and the drop shape was digitized. Surface tension was measured for the solution as a function of temperature. All surface tension measurements were conducted at a pressure of 72 psi.

## **2.7. Experimental Procedure for HPHT Pendant Drop**

1. Fill tank A with liquid while valve “A” closed, Figure 3.
2. Back off the pressure generator (PG1), to fill the (PG1), while “A” is open.
3. Close valve “A”, closed (PG1), however “D” closed and “C” open.
4. Keep turning (PG1) until only liquid comes out from the capillary without air, then close “C”.
5. Heat the system to the test temperature, maximum temperature that can be reached is 390°F.

6. Open “C”, and rotate “PG1”, until liquid comes out from the capillary.
7. Open “E”, apply N<sub>2</sub> (50-100 psi) from line “B”, (for ~3 minutes).
8. Close “E”, open “F”, back off (PG2) up to the middle, then let some Nitrogen vent through T2 by opening “G”.
9. Close “G” and apply pressure through “B” until the test pressure.
10. When you opening “C” rotate (PG1), until few drop coning from the capillary (~5 drops), then start taking the reading.
11. Repeat (9), when ever pressure needs to be change.
12. If condensate start formed on the glass window closed “F” and let the system vent through “E” slowly, “E” has to be open.
13. To increase the pressure higher than the Nitrogen coming from the supply, use (PG2) to increase the pressure, “F” is open.



**Figure 3:** Flow sheet of PD-E700LL-H

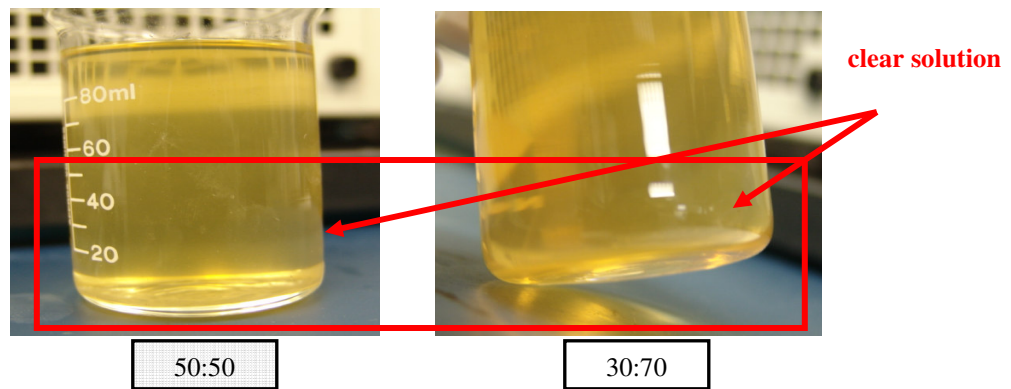
## CHAPTER III

### FLUIDS COMPATIBILITY

#### 3.1. Drilling Fluid Filtrate (DFF)

##### *3.1.1 DFF Mixed with Ethyl Lactate Ester at Room Temperature*

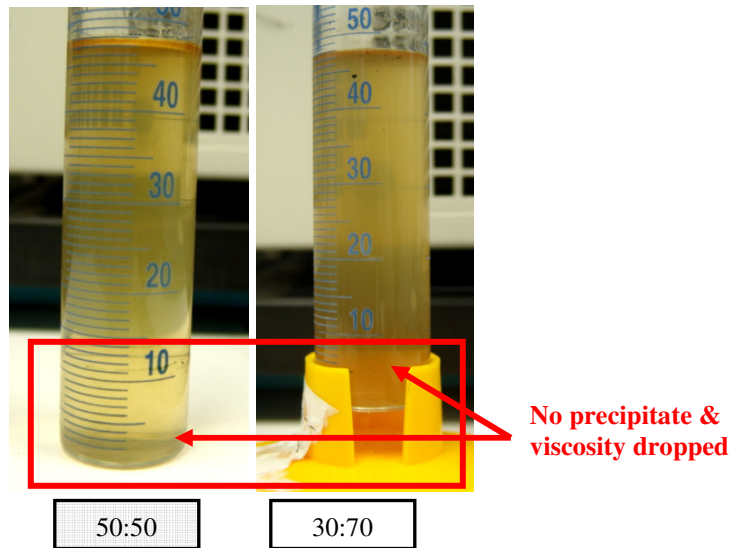
The HPHT filter press instrument was utilized to filter the liquid part (filtrate) from the formate based drilling fluid and make it a solids-free fluid. The objective behind this step was to simulate the actual field operation since the liquid phase only entered the formation while most of the solids were retained on the filter cake layer. At the beginning, ethyl lactate ester was selected because it showed a great dissolving efficiency in removing filter cake damage that was initiated by another water-based mud system (Al Otaibi et al., 2006; Nasr-El-Din et al., 2005). A 10 vol.% of ethyl lactate ester solution was mixed with drilling fluid filtrate at a ratio of 50:50 and at room temperature, Figure 4. The viscosity of the filtrate solution was dropped after mixing with the ester, and no precipitate was noticed for 24 hours. The ratio was changed in the second test to be 30:70, between drilling fluid filtrate and the ester solution, Figure 4. The same behavior was noticed, and for that reason no further tests were performed. Filtrate of drilling fluid was expected to compose of potassium formate brine, polyanionic cellulose (PAC), XC-polymer and calcium carbonate particles. The ester solution, however, will not decompose to alcohol and lactic acid at room temperature.



**Figure 4:** Compatibility of DFF with lactate ester at room temperature

### 3.1.2 DFF Mixed with Ethyl Lactate Ester at 300°F

The same samples in Figure 4 were loaded in the HPHT cell to examine their behavior at reservoir temperature, 300°F. The samples were left for one day in the cell, and no precipitate or phase separation was observed as shown in Figure 5. The generated alcohol and lactic acid at high temperatures did not cause any precipitate with drilling fluid filtrate. The mixing process should be done gradually while increasing the solution temperature but it is too difficult to maintain that without applying a pressure to avoid any vaporization. The viscosity of the solution was clearly dropped compared to the room temperature sample.



**Figure 5:** Compatibility of DFF with lactate ester at 300°F

### 3.1.3 DFF Mixed with Formation Brine at Room Temperature

Formation brine recipe of Unyazah reservoir (gas reservoir) was requested from the field engineer and then a synthetic brine sample was prepared in the lab at room temperature, Table 4. Drilling fluid filtrate was mixed with the formation brine at five different mixing ratios and at room temperature. Table 5 shows the five mixing ratios that were used and the observation at room temperature. All five solutions showed a solid precipitate, and a phase separation after 30 minutes of the mixing time as shown in Figure 6. The tests at higher temperatures were cancelled because all samples were precipitated and also separated at room temperature.

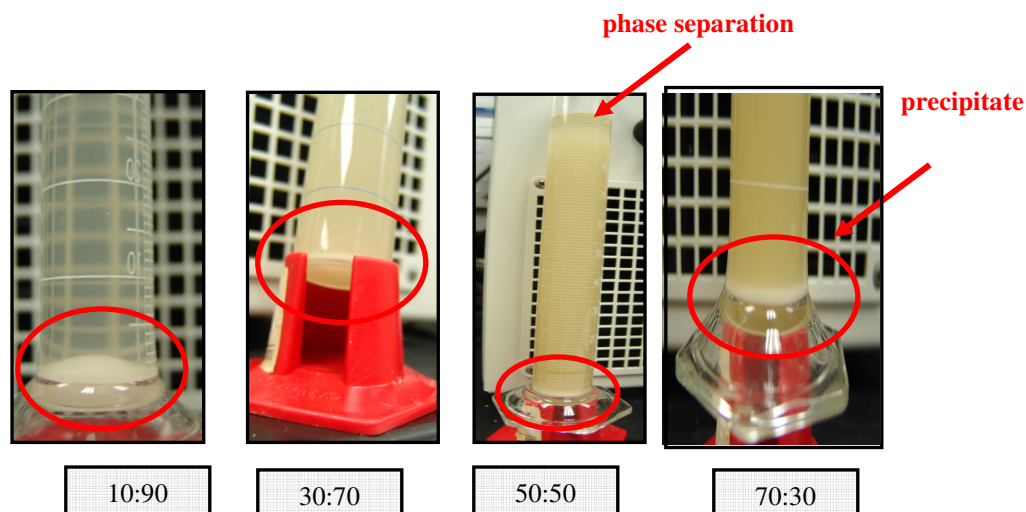
**Table 4:** Unyazah formation brine composition and ions concentration

Compound	Concentration, g/l
NaCl	142.57
CaCl <sub>2</sub> .2H <sub>2</sub> O	76.759
MgCl <sub>2</sub> .6H <sub>2</sub> O	9.8259
Na <sub>2</sub> SO <sub>4</sub>	0.1479
NaHCO <sub>3</sub>	0.7889

Ions, mg/l								pH	Density, lb/ft <sup>3</sup>
Na	Ca	Mg	SO <sub>4</sub>	Cl	CO <sub>3</sub>	HCO <sub>3</sub>	TDS		
56,356	20,926	1,175	100	126,935	0	573	206,065	6.031	70.86

**Table 5:** Five mixing ratios used at room and high temperatures

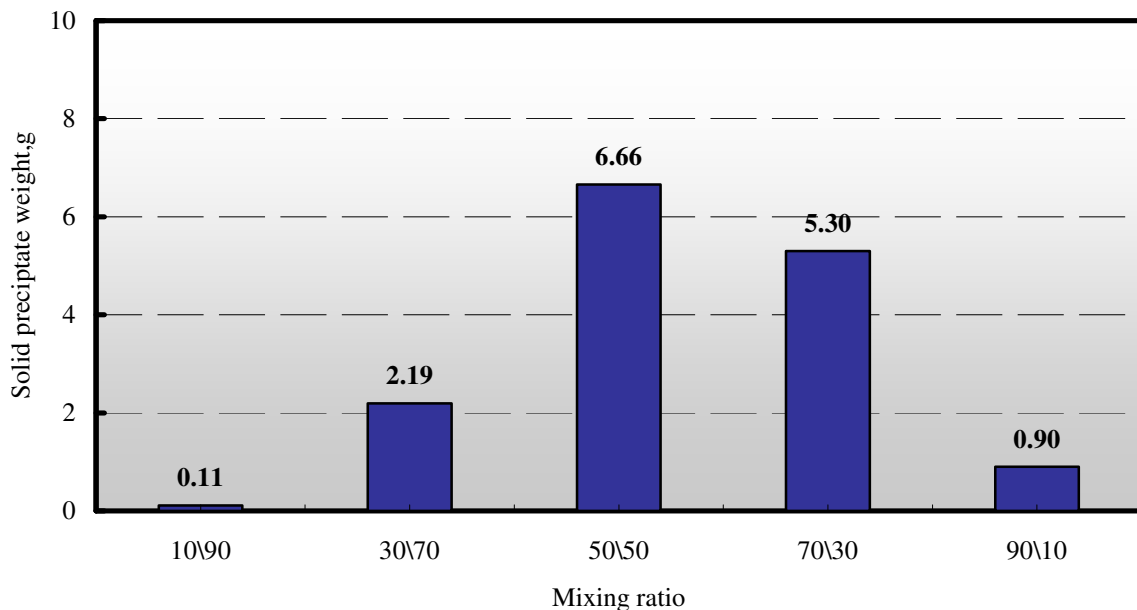
	Drilling fluid filtrate : formation brine				
<b>Mixing ratio</b>	10:90	30:70	50:50	70:30	90:10
<b>Observation</b>	Phase separation and different amounts of solid precipitate				

**Figure 6:** Compatibility of DFF with formation brine at room temperature

### 3.2. Solids-free Potassium Formate Brine (PFB)

#### 3.2.1 PFB Mixed with Unyazah Formation Brine (UFB) at Room Temperature

The used potassium formate brine (PFB) in all experiments was received from the field, and the pH and the density were measured at room temperature and equal to 8.05 and 95.83 pcf, respectively. Compatibility tests between PFB and UFB were tried first at room temperature and five different mixing ratios. The resulted solid precipitates in all new solutions were filtered by using filter paper size of 1.2  $\mu\text{m}$ . The largest amount of precipitate was noticed at ratio of 50:50 between PFB and UFB. The weight of the precipitate as a function of mixing ratio is shown in Figure 7. The solid precipitate for the first sample (10:90) was almost negligible (0.11 g). All mixtures were cloudy during and after the mixing, and clear precipitate was also noticed for all samples except the one at 10:90.



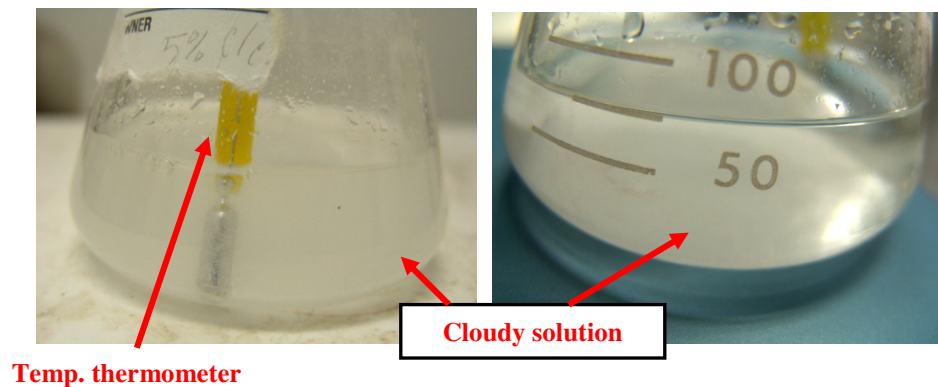
**Figure 7:** Compatibility tests between PFB and UFB at room temperature



### 3.2.2 PFB Mixed with UFB at Temperatures 113-127°F

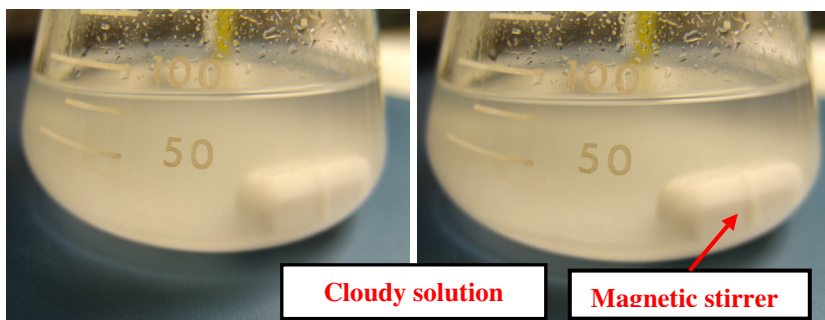
Some authors discussed the solid precipitate concern while mixing PFB with formation brine, and they said the precipitate was only noticed at room temperature and not at reservoir conditions (Downs et al., 2005; Byrne et al., 2002). Based on that, a new set of compatibility tests between PFB and UFB were conducted but at higher temperatures. A hot plate was utilized to heat up the UFB in a glass beaker and then PFB was added slowly to it. The temperature ranges were between 113 and 127°F before and during the mixing. A magnetic stirrer was used for agitation and a temperature thermometer was used for measuring the temperature manually.

Five different mixing ratios were used as done in the previous step. As noticed in room temperature tests, the largest solid precipitates were observed after mixing PFB with UFB at ratio 50:50, Figure 8.



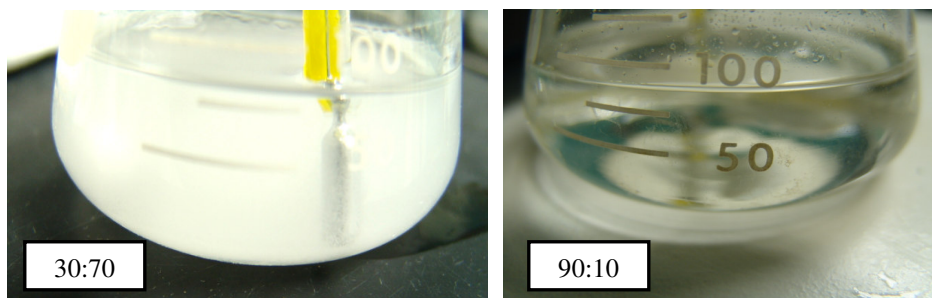
**Figure 8:** Compatibility of PFB with UFB at ratio of 50:50

The amount of solids precipitates was decreased as the fluid ratio changed to 70:30 as shown in Figure 9. During the mixing of the solution, the temperature range was between 113 and 122°F.



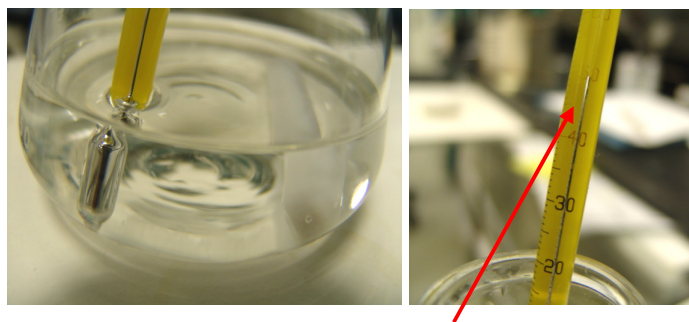
**Figure 9:** Compatibility of PFB with UFB at ratio of 70:30

Fewer amounts of precipitates were noticed as the fluid ratios of PFB and UFB changed to 30:70 and 90:10, Figure 10. The temperature range for the 30:70 test was 113–118°F. It was difficult to maintain the same temperature degree for all tests because the hot plate temperature in the lab was affected by the air condition. The solution temperature was also affected by PFB temperature before added to the UFB solution. In the second test (90:10), the temperature range was between 113 and 127°F.



**Figure 10:** Compatibility of PFB with UFB at ratios of 30:70 and 90:10

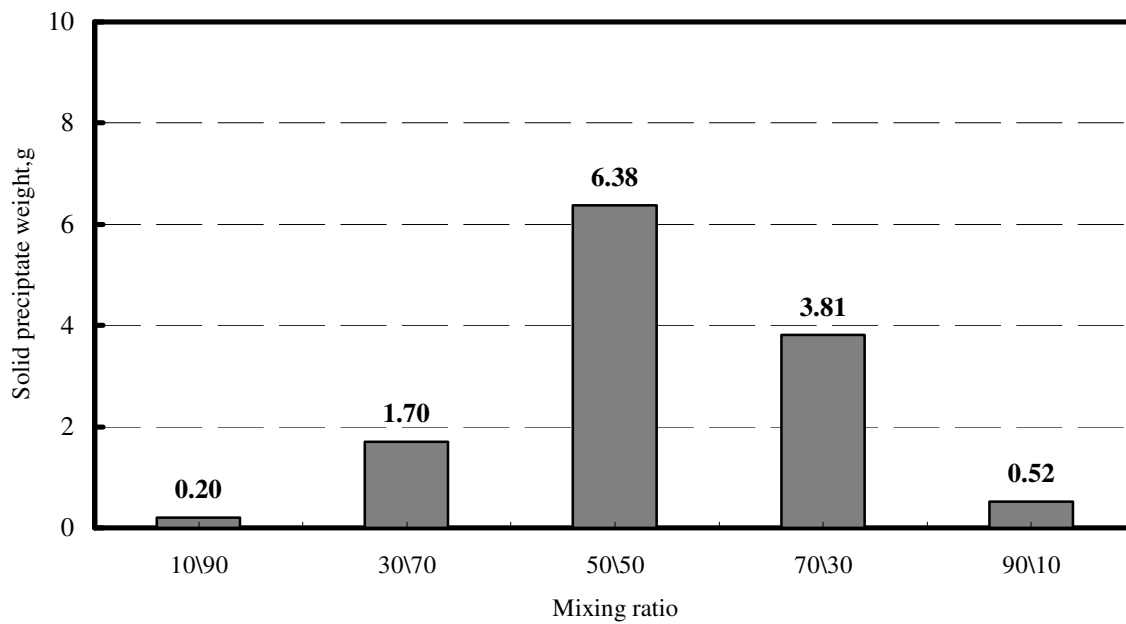
In the last mixture, PFB was decreased to 10 ml while the volume of UFB was increased to 90 ml. The result was a clear solution and no precipitates had been noticed during and after the mixing, Figure 11.



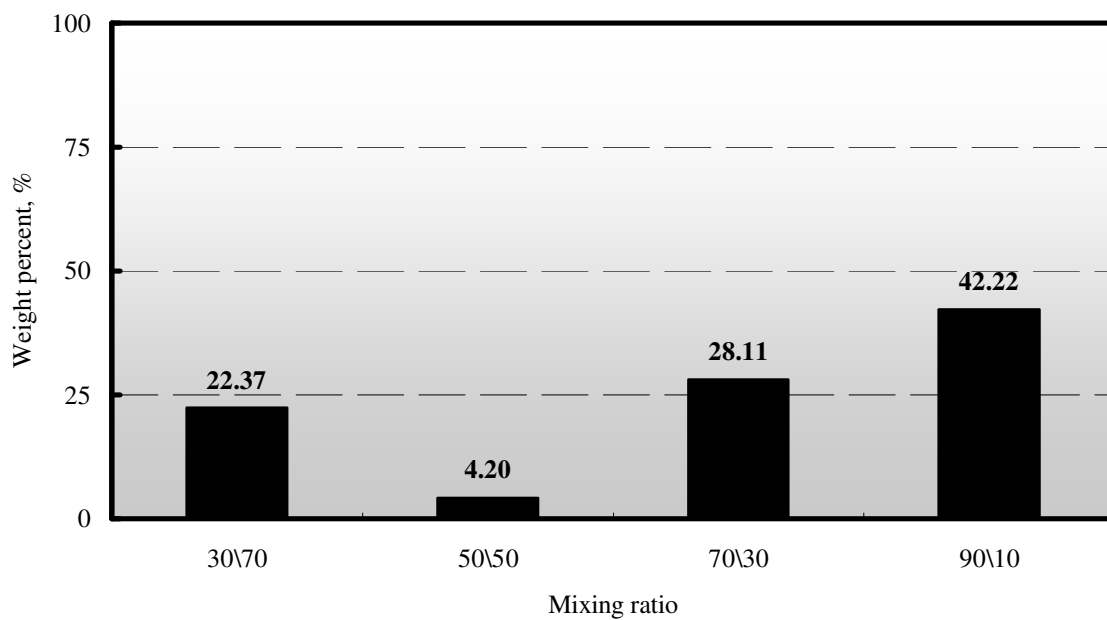
**Temperature = 50°C = 122°F**

**Figure 11:** Compatibility of PFB with UFB at ratio of 10:90

Figure 12 shows the weight of the solids which were precipitated as a result of mixing PFB with UFB at a wide temperature range (113-127°F). The solid precipitates at room temperature are greater than what was observed at temperatures 113 to 127°F except at one ratio (10:90), Figure 13. The small amount of precipitate that was noticed for the 10:90 mixture at both conditions was almost negligible and may be due to the contamination of the glass beaker and filtration kit. For that reason, it will not be considered in the following comparison. As temperature increased, more salt will be dissolved in the solution and that confirms the compatibility results at room and high temperatures.



**Figure 12:** Compatibility tests between PFB and UFB at temperature range of 113-127°F



**Figure 13:** The weight difference of the solid precipitate at room and higher temperatures

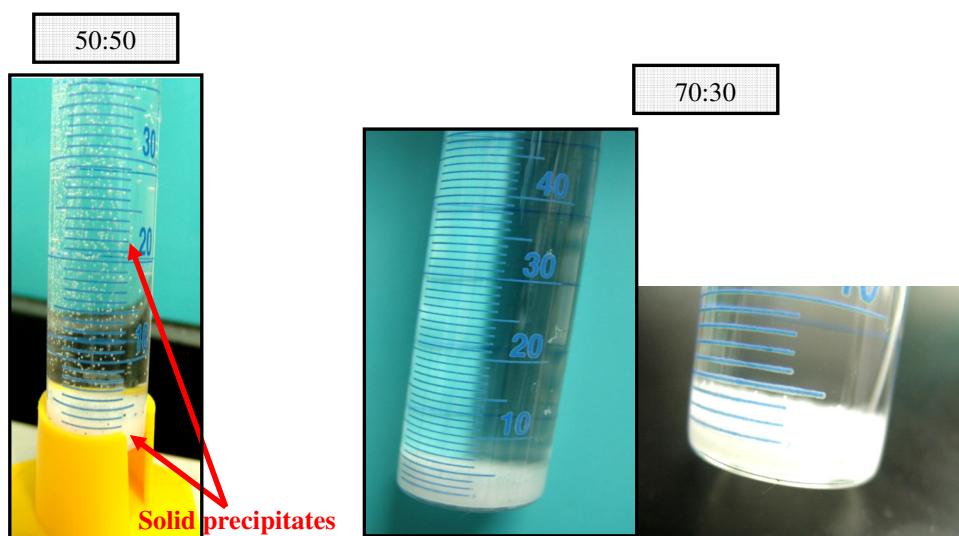
### 3.2.3 PFB Mixed with UFB at Temperatures 273-297°F

At reservoir temperature, it was difficult to conduct the test without applying enough pressure to avoid any fluid vaporization. For that reason, a new set of mixtures were prepared at temperatures 122-140°F and were reused at higher temperatures. They were loaded immediately in HPHT visual cell at temperatures 273-297°F and pressure 250-300 psi. All samples were left in the cell for 21.5-23 hours.

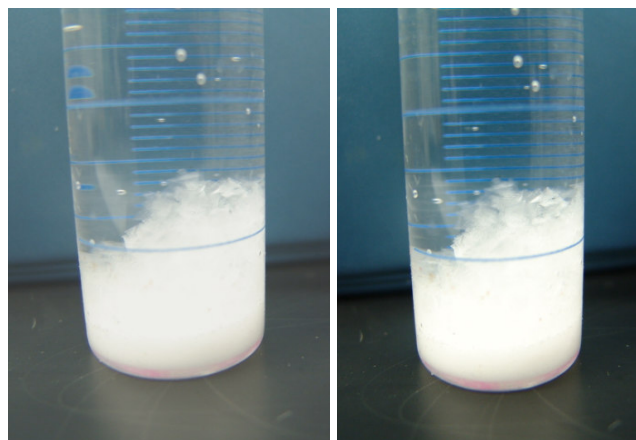
The first sample (50:50) was mixed at 122°F and at atmospheric pressure as done in previous tests. The mixture was then loaded in the HPHT cell at temperature of 289°F and pressure of 300 psi, Figure 14. The cell was disconnected after 21.7 hours and the temperature was reduced to room temperature using the control button. A solid precipitate was noticed in the mixture before loading the sample. Decreasing the solution temperature caused more precipitations to settle down. The solution at high temperature was supersaturated and it precipitated as the temperature decreased.

The next sample is 70:30 PFB/UFB solution that was prepared first at 138°F and it was cloudy and had some precipitate. Then, the sample was loaded directly in HPHT cell at 286°F and 250 psi. The cell was disconnected to remove the sample after 23 hours of soaking, Figure 14.

More solids precipitates were noticed as the ratio changed to 30:70 of PFB/UFB. The sample was first prepared at 140°F and then loaded in HPHT cell at 273°F for 21.3 hours, Figure 15.

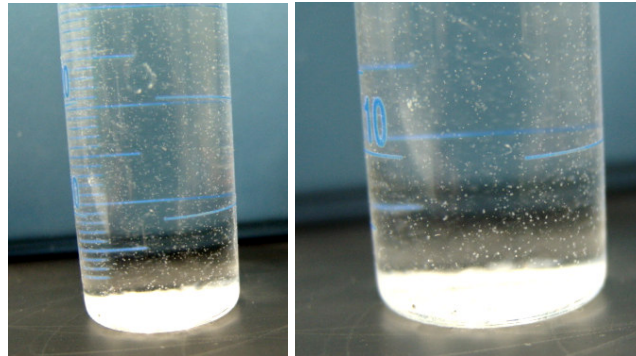


**Figure 14:** Compatibility of PFB with UFB at two different ratios



**Figure 15:** Compatibility of PFB with UFB at ratio of 30:70

Less amount of solid precipitate was observed in the 90:10 PFB/UFB mixture. The sample was prepared at 133°F before loading in the HPHT cell at 289°F. The cell was disconnected and the sample had some precipitate as shown in Figure 16.



**Figure 16:** Compatibility of PFB with UFB at ratio of 90:10

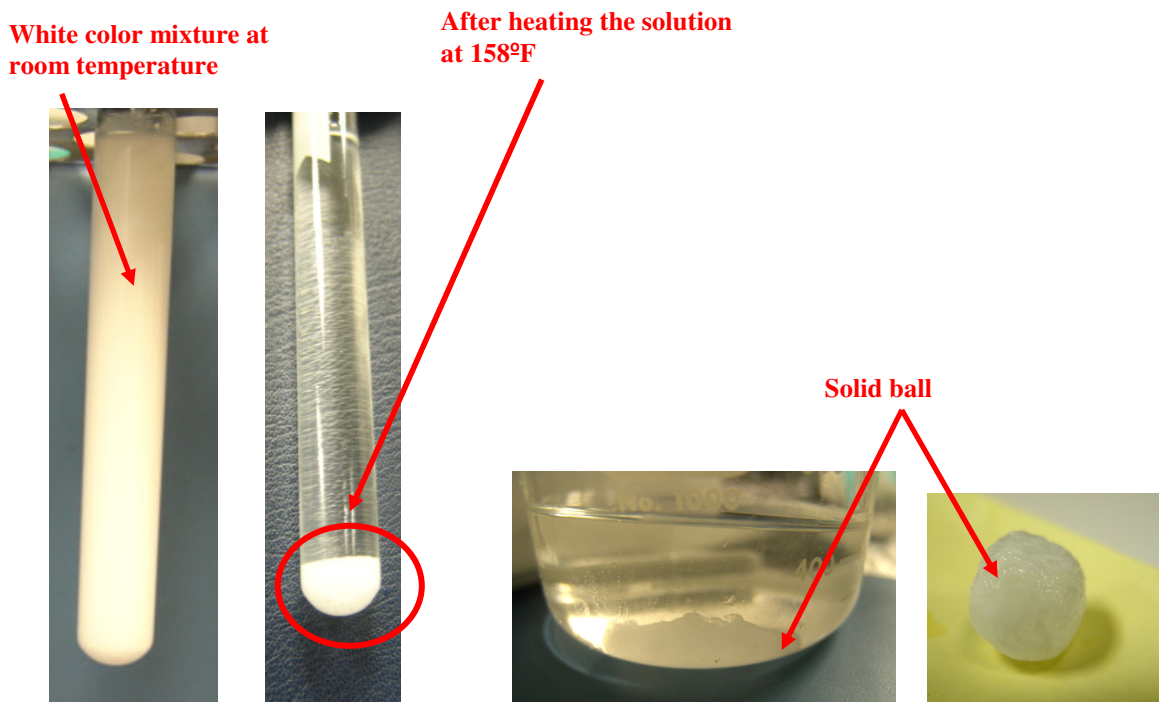
The weight of the solid precipitate for 10:90 PFB/UFB mixture was measured and it was the lowest compared to others mixtures. The solution was prepared first at 122°F and then loaded in the cell at 297°F. The solid precipitate weight was measured and it will not be mentioned since the mixing method was not performed at reservoir temperatures and also the results were unreliable.

### **3.3. Organic Acids Prepared in PFB Solution**

#### *3.3.1 10 wt% Citric Acid Mixed with PFB*

A 10 wt% of citric acid was prepared in 90 pcf PFB at room temperature. It was shown by others that citric acid has the highest dissolving efficiency to the filter cake damage, which was generated by formate based fluid, compared to other breakers (Svendsen et al., 1995). The citric acid was mixed for 3 hours and a white precipitate was noticed

during the mixing at low acid concentration. The color of the mixture changed with time to a white color. The white precipitate changed to a big solid ball during the mixing. The fluid was filtered by using 1.2  $\mu\text{m}$  filter paper and a thick layer of white precipitate was noticed on the paper. It means that citric acid was not compatible with PFB solution at room temperature as shown in Figure 17.



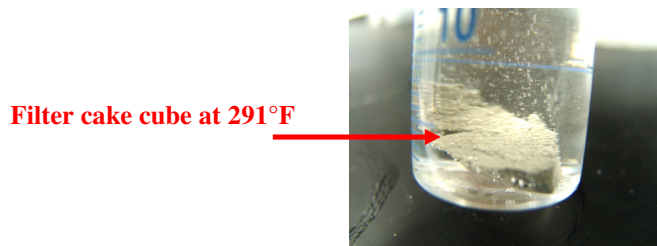
**Figure 17:** Compatibility of 10 wt% citric acid with PFB at room temperature

A 10 wt% citric acid was prepared in PFB solution but at 158°F and at atmospheric pressure. No solid precipitates were noticed during and after the mixing. The same sample was loaded in the HPHT visual cell to examine the fluids compatibility at reservoir temperature. The cell temperature and pressure were 298°F and 200 psi,



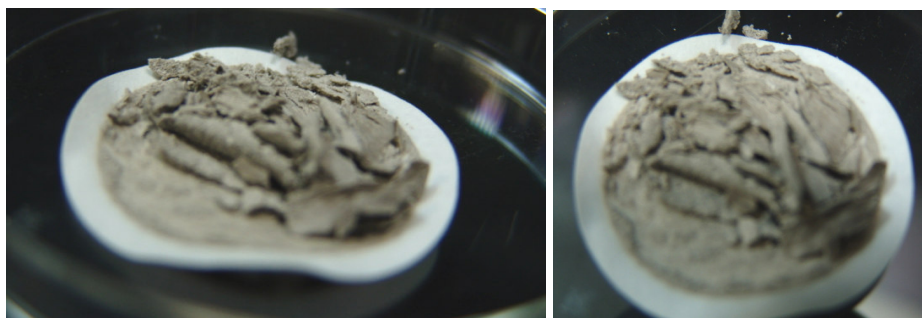
respectively. The mixture was clear and no precipitates were observed during and after 21.5 hours of soaking.

A small cube of filter cake layer was removed from a damaged ceramic disk and placed in a graduated cylinder full of 10 wt% citric acid solution, Figure 18. The objective of this step was to examine the effect of the acid solution on filter cake damage and to observe any chemical reaction at high temperatures. The cell pressure was fixed at 230 psi and the reaction was started when the cell temperature increased to 291°F. At the beginning, small bubbles were released and that was a good indication that the acid reacted with the uncovered calcium carbonate particles with biopolymers.



**Figure 18:** Effect of 10 wt% citric acid on filter cake

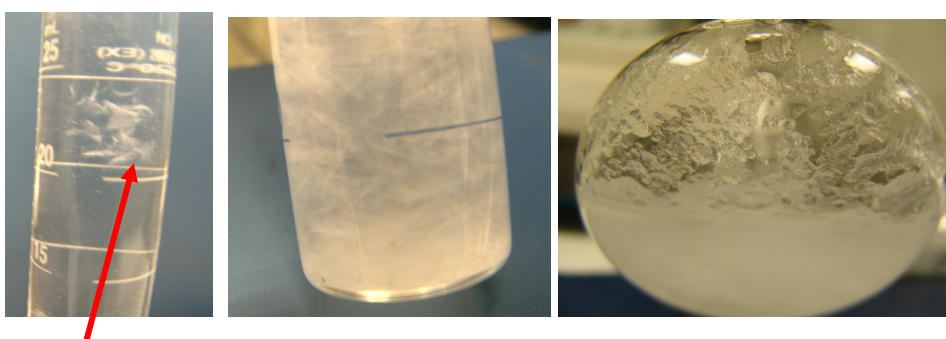
The weight of the filter cake sample was recorded before and after the soaking. The result showed only 10% of the filter cake was dissolved by citric acid. The filter cake solid after reaction was filtered as shown in Figure 19.



**Figure 19:** Filter cake solids after the reaction with 10 wt% citric acid

### 3.3.2 10 wt% Formic Acid Prepared in PFB Solution

Formic acid at a concentration of 10 wt% was prepared in PFB at room temperature. Some solid precipitates were suspended in the solution as shown in Figure 20. The sample was left for 30 minutes at room temperature and then it became a gel. Larsson and Nahrungbauer discussed in their article that potassium hydrogen diformate was prepared in the lab by dissolving potassium formate in formic acid during heating (Larsson and Nahrungbauer, 1968). The mixture was heated to 158°F and it returned to be a liquid with some solids, Figure 21.

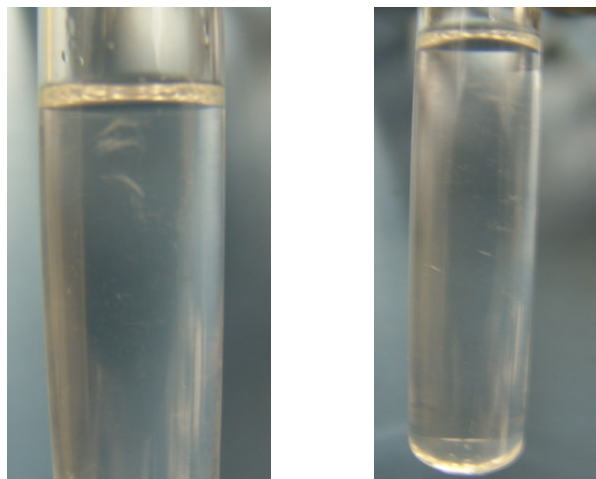


**Suspended solids at room temperature**

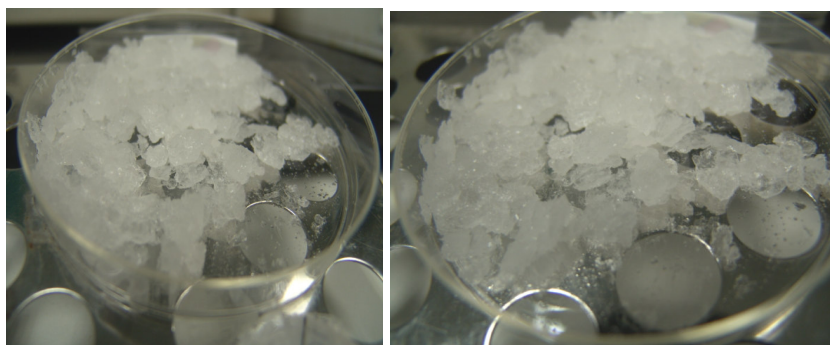
**Gel fluid at room temperature**

**Figure 20:** 10 wt% formic acid prepared in PFB solution at room temperature

The precipitated solids were similar to salt crystals; they were filtered by using 1.2  $\mu\text{m}$  filter paper, Figure 22.

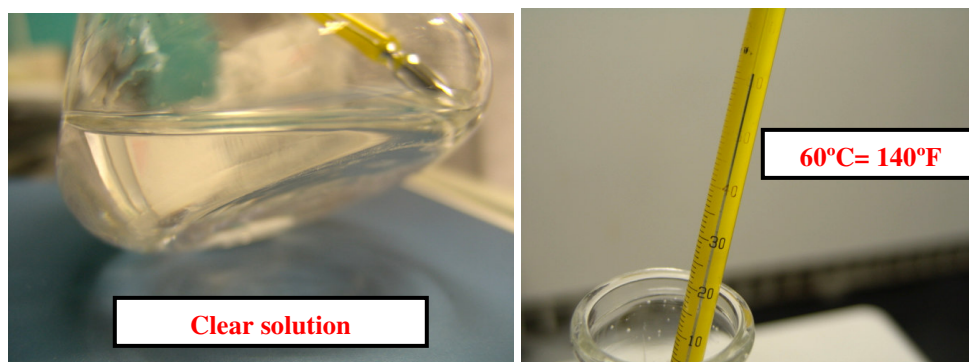


**Figure 21:** 10 wt% formic acid prepared in PFB solution at 158°F



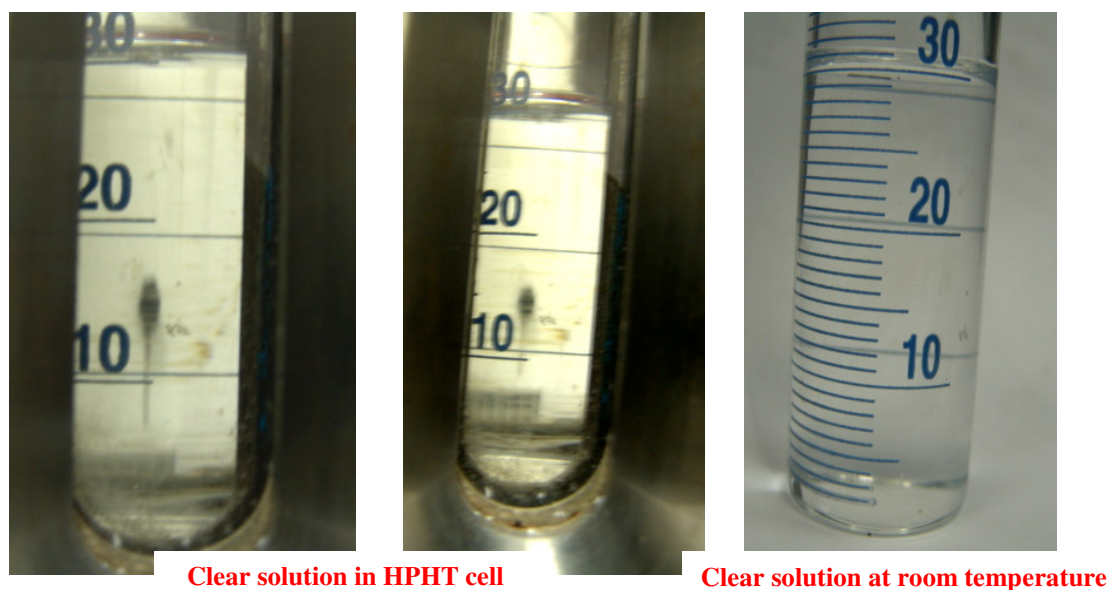
**Figure 22:** Solids filtered from the 10 wt% formic acid solution that prepared at room temperature

The 10 wt% formic acid solution was prepared at higher temperatures using a hot plate. The mixture was clear with no precipitates as shown in Figure 23. The temperature condition during the mixing was 140°F.



**Figure 23:** 10 wt% formic acid prepared in PFB solution at 140°F

The mixture was loaded in the HPHT cell for 2 hours at reservoir temperature (289°F) and at 200 psi, Figure 24. No precipitates were noticed at that temperature and also when the cell was disconnected to remove the sample. As noticed before, the sample became a gel when it cooled down to room temperature after 30 minutes.



**Figure 24:** 10 wt% formic acid prepared in PFB solution at 289°F

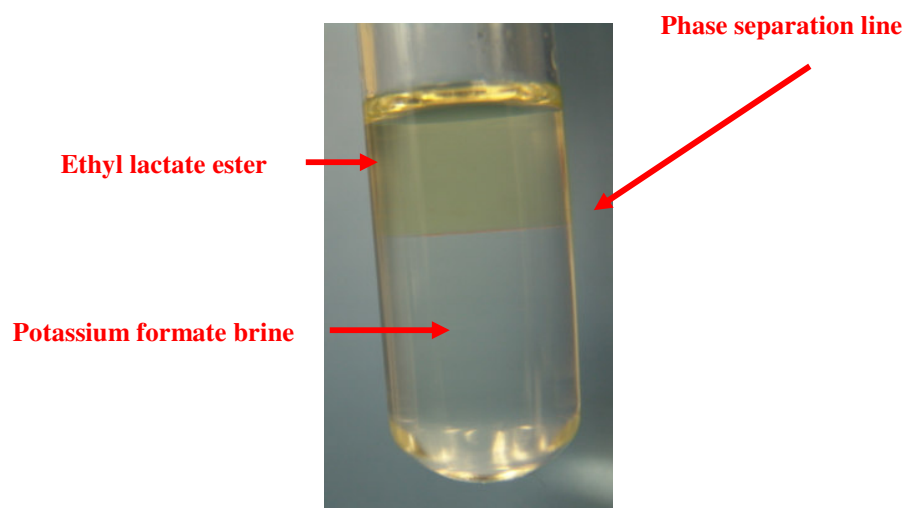
### *3.3.3 10 wt% Acetic Acid Prepared in PFB Solution*

Acetic acid was left to the end since it is a weak acid compared to formic and citric acids. A 10 wt% of acetic acid was prepared in PFB at room temperature and a good compatibility was noticed between the two fluids. The mixture was filtered through 1.2  $\mu\text{m}$  filter paper to make sure no precipitates were existed. The same solution was prepared at 140°F and it was compatible during and after the mixing. At a higher temperature (255°F), no precipitates were noticed for 20 hours. Acetic acid was the only compatible acid with PFB at room and high temperatures compared to other acids.

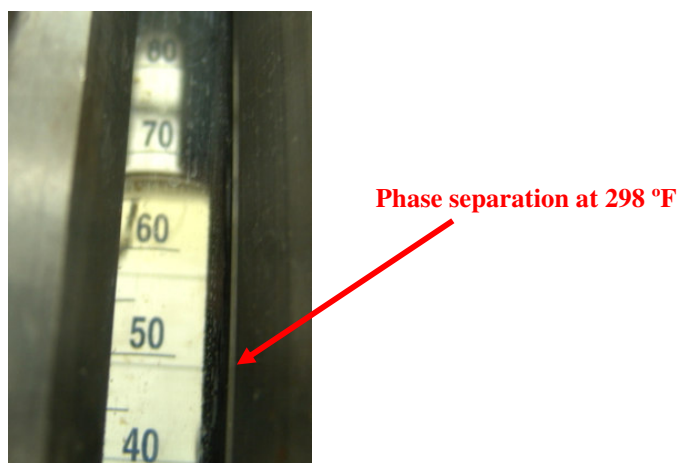
### *3.3.4 5 and 10 wt% Ethyl Lactate Ester Prepared in PFB Solution*

In section 3.1, compatibility of ethyl lactate ester was tested with potassium drilling fluid filtrate and it was compatible. Ester solution has been used lately to remove filter cake damage since it generates lactic acid as temperature increases with time (Moses and Harris, 2004). The ethyl lactate ester solution should be prepared in formate brine to increase the fluid density and also to use similar brine to the one in the drilling fluid.

Ethyl lactate ester was prepared first at room temperature and at a concentration of 10 wt%. The ester solution showed a phase separation with PFB, Figure 25. The same mixture was re-prepared at 158°F and atmospheric pressure and the same separation was noticed. The prepared solution at 158°F was loaded in HPHT visual cell at temperature and pressure of 298°F and 300 psi, Figure 26. The mixture was kept in the cell for 20 hours.



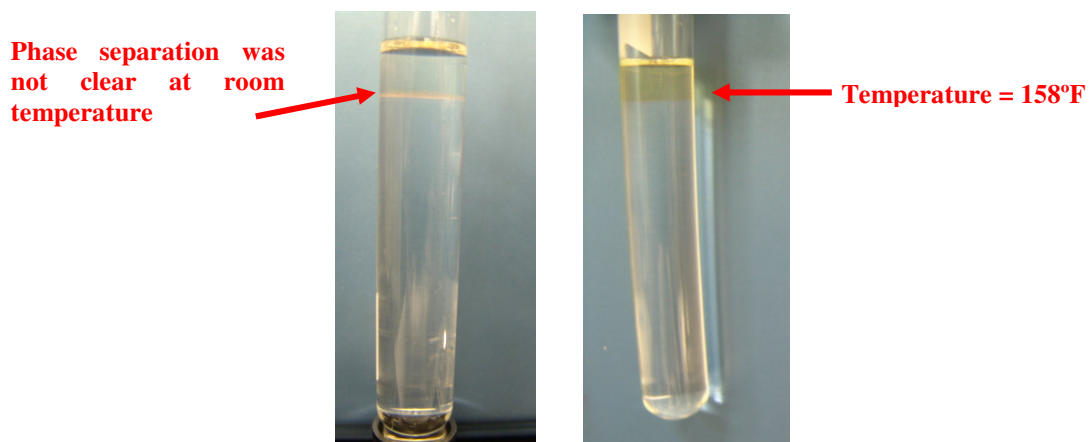
**Figure 25:** 10 wt% ethyl lactate ester prepared in PFB solution at room temperature



**Figure 26:** 10 wt% ethyl lactate ester prepared in PFB solution at 298°F

The concentration of ethyl lactate ester was reduced to 5 wt.% and the mixtures were prepared in PFB at the same conditions. At room temperature, the phase separation was noticed and the ester was incompatible with PFB. Then, the mixture was heated to 158°F

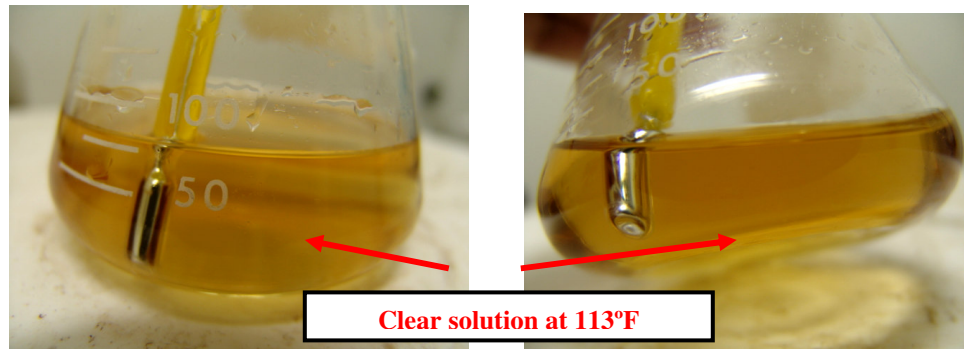
and the observation was similar to the room temperature test result. The phase separation was clear as temperature increased from room temperature to 158°F, Figure 27.



**Figure 27:** 5 wt% ethyl lactate ester prepared in PFB solution prepared at room temperature

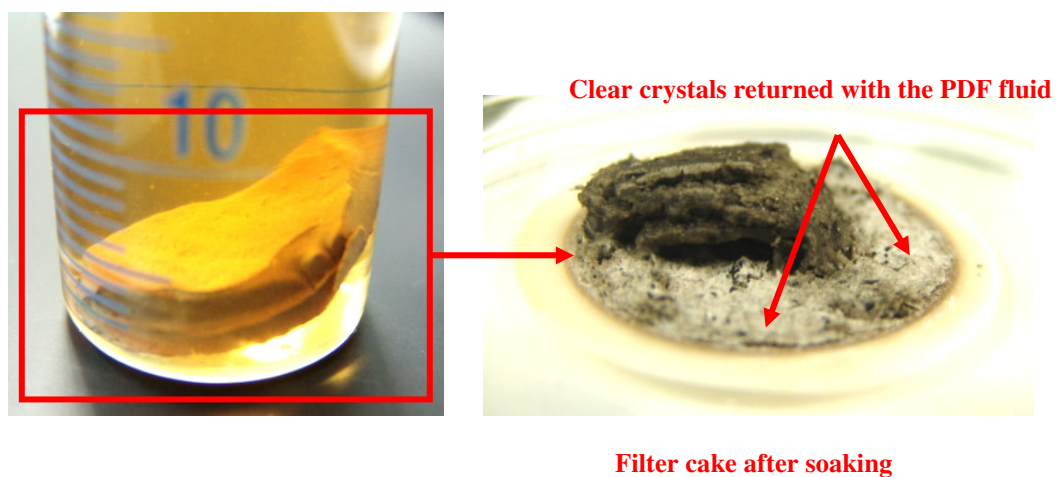
### 3.3.5 Chelating Agent (DTPA) Prepared in PFB Solution

DTPA (Diethylenetriaminepentaacetic acid) was prepared in PFB at a ratio of 50:50; no precipitate or phase separation was noticed at room temperature. The DTPA was added to the PFB at 113°F and it was compatible, Figure 28. The same fluid was loaded in HPHT cell at 284°F and pressure of 200 psi. The sample was left in the cell for 24 hours. Small crystals were noticed after filtering the solution through 1.2  $\mu\text{m}$  filter paper.



**Figure 28:** DTPA prepared in PFB solution at a ratio of 50:50

To examine the effect of the DTPA on the filter cake damage, a small portion of the filter cake layer was extracted and then placed in a 100 ml graduated cylinder. The cylinder was loaded in the HPHT visual cell at a temperature and pressure of 284°F and 200 psi, respectively. The cake sample was soaked in the DTPA solution for 27 hours. The cell was disconnected and the solution was filtered through 1.2  $\mu\text{m}$  filter paper. The chelating agent fluid was ineffective to dissolve the calcium carbonate particles because it was coated with biopolymers, Figure 29.



**Figure 29:** Effect of DTPA on filter cake



### 3.4. Xanthan and Cellulose Enzymes Prepared in PFB Solution

The drilling fluid recipe that was used in the field had cellulose and xanthan polymers to reduce the fluid losses into the formation and to carry the drilling cuttings to the surface. Filter press experiments gave insufficient removing efficiency when organic acids were used to remove the filter cake damage as can be seen in Chapter IV. The xanthan and cellulose polymers were the main reason because they were covering the calcium carbonate particles in filter cake. Weak acids cannot dissolve or remove that polymer layer.

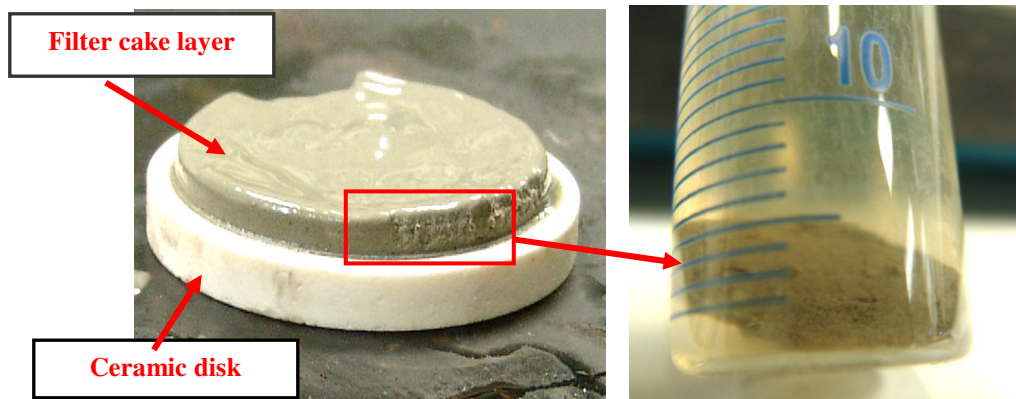
Enzymes have been used commonly to break down the polymer chains and hydrolyze it to small sugar units. Two classes of enzymes were received from a local service company and were used at a concentration of 10 wt%. The enzymes were prepared in potassium formate brine at room temperature first and were compatible. The solution was prepared at 149°F to examine the fluid compatibility at higher temperatures as shown Figure 30.

Xanthan and cellulose  
enzymes in PFB at 149°F



**Figure 30:** 10 wt% of xanthan and cellulose enzymes prepared in PFB solution

A simple test was conducted in the HPHT visual cell by taking a small portion of the filter cake layer as shown in Figure 31. The graduated cylinder that is usually loaded in the visual cell was half filled with enzymes solution. The reason for that step was to ensure full fluid coverage to the filter cake sample. After that, the sample was placed in the cell and connected. The temperature and pressure of the cell were 284°F and 200 psi for 23 hours. The test result showed the acid was needed with the enzyme solution to dissolve the calcium carbonate particles.



**Figure 31:** Effect of 10 wt% of xanthan and cellulose enzymes on the filter cake

## CHAPTER IV

### RESULTS AND DISCUSSION

#### 4.1. Analysis of Fluids Compatibility Tests

##### *4.1.1 DFF Mixed with UFB at Room Temperature*

X-Ray powder Diffraction (XRD) and ESEM techniques were utilized to determine the nature of the solids precipitates that were collected on the filter papers. The solids were precipitated as a result of mixing potassium formate drilling fluid filtrate and Unayzah formation brine at different volume ratios. At the beginning, only three samples of drilling fluid filtrate (DFF) and Unayzah formation brine (UFB) were mixed at ratios of: 10:90, 30:70 and 50:50.

##### *4.1.1.1 10% DFF Mixed with 90% UFB (Sample No. 1)*

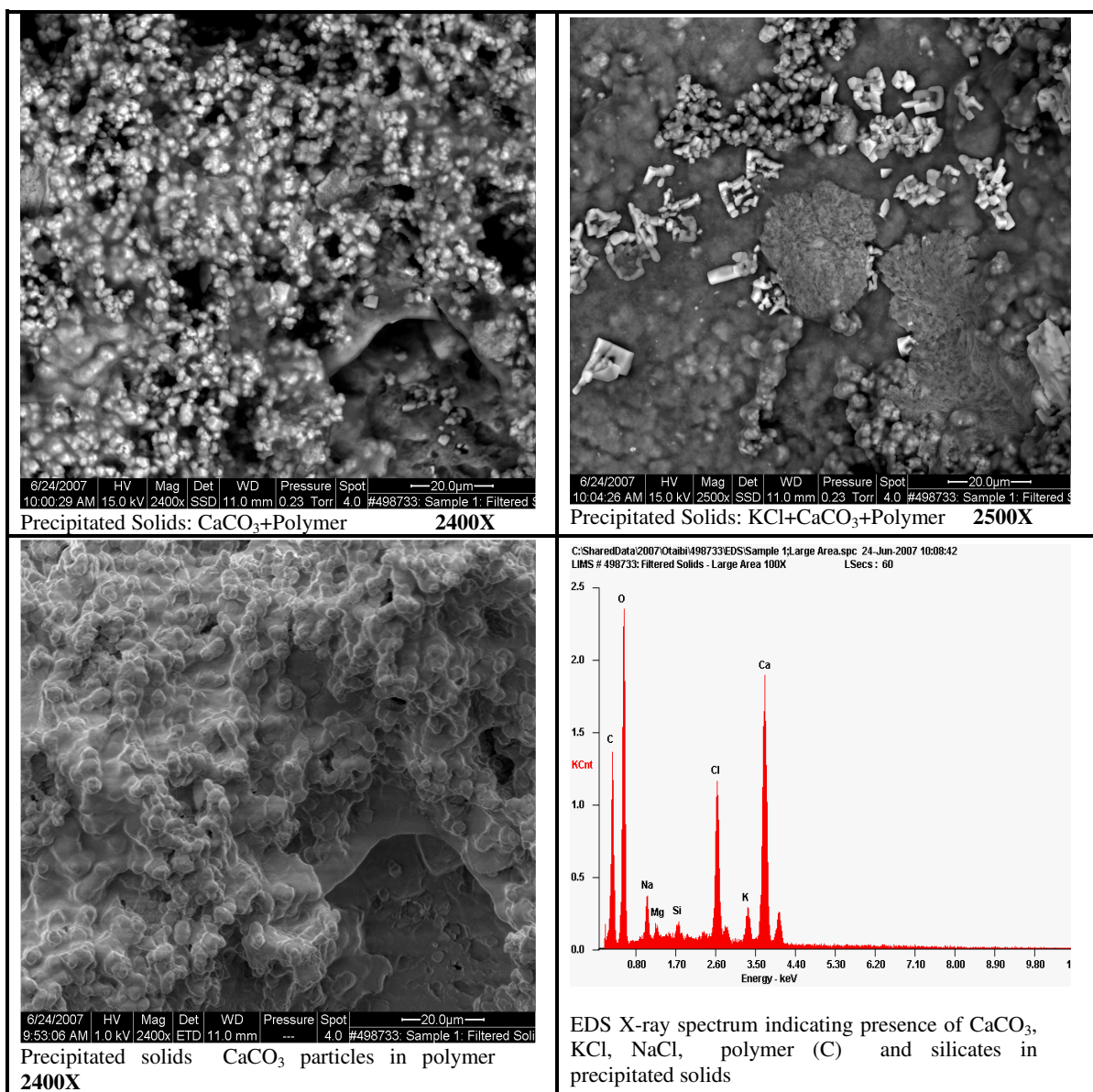
A 10% of DFF was added slowly to 90% UFB and it was stirring during mixing to ensure proper mixing method. The XRD result of the precipitate shows halite (NaCl) is a major component in the sample. The source of the halite is coming from the formation brine. Sylvite (KCl) and calcite ( $\text{CaCO}_3$ ) are noticed as minor components in the sample. Calcite was used as a drilling fluid additive while drilling the target zone and that is the main source. Calcium formate- $\text{C}_2\text{H}_2\text{CaO}_4$  is also present in the sample.

Figure 32 shows the images and EDS X-ray spectrum of the precipitated solids on a filter paper. The secondary and backscattered electron images showed spherical  $\text{CaCO}_3$  particles that are dispersed in a polymer matrix. The  $\text{CaCO}_3$  particles are about 2

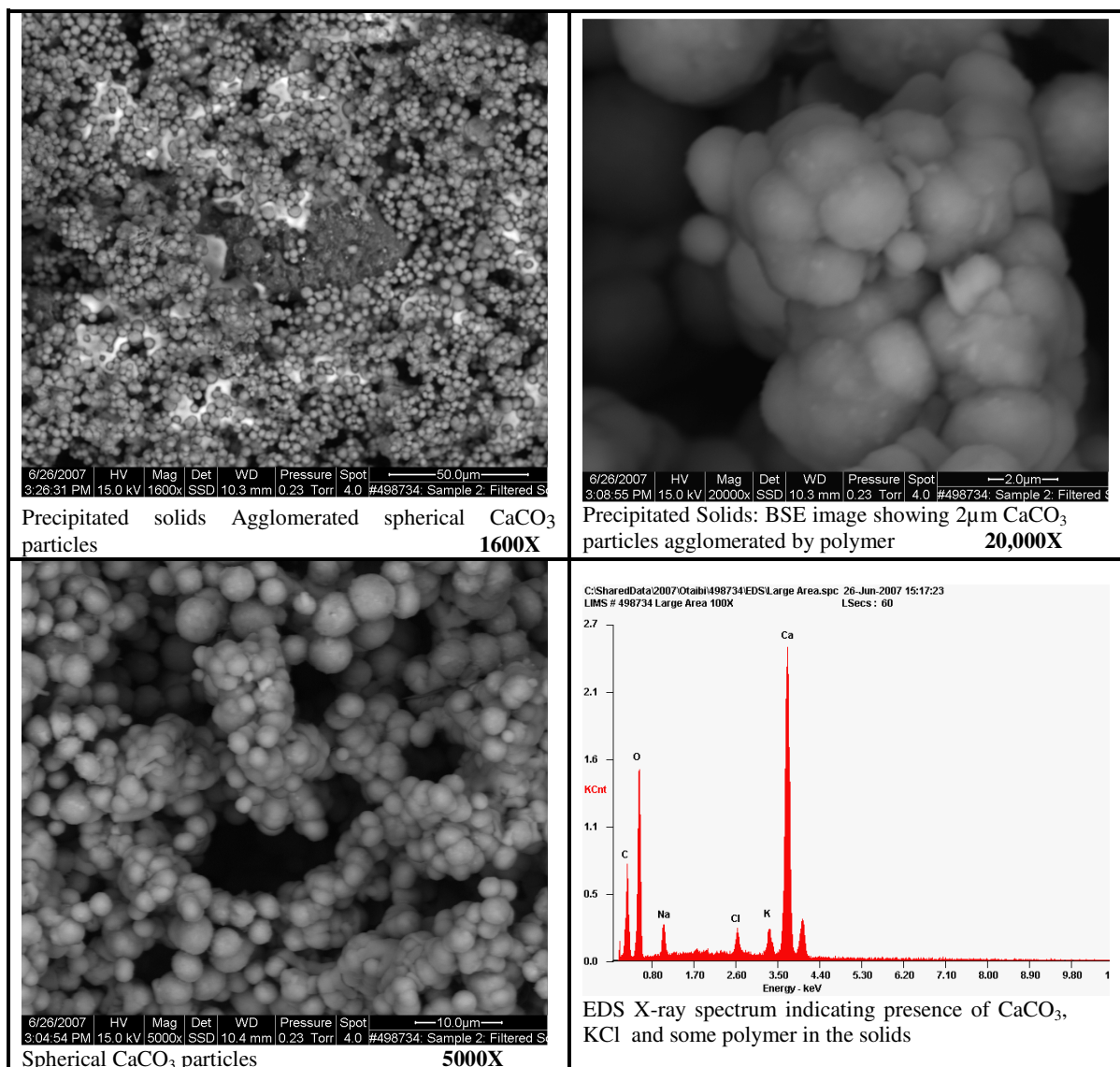
$\mu\text{m}$  in diameter and appeared to have been agglomerated by polymers in the fluid. Both ESEM and XRD show presence of NaCl, KCl and small amounts of calcium formate and quartz in the precipitate. The source of the quartz is from the formation since the used drilling fluid is a field sample.

#### *4.1.1.2 30% DFF Mixed with 70% UFB (Sample No. 2)*

The XRD result shows calcium carbonates (calcite and vaterite ( $\text{CaCO}_3$ )) are the major component of the sample. Calcium formate- $\text{C}_2\text{H}_2\text{CaO}_4$  is also present in the sample. Figure 33 shows the images and EDS X-ray spectrum of the precipitated solids. The ESEM images show mainly spherical  $\text{CaCO}_3$  particles with some KCl, NaCl and silicate particles. The  $\text{CaCO}_3$  particles are about  $2 \mu\text{m}$  in diameter and appeared to be coated with polymer. The spherical shape and ( $\sim 2 \mu\text{m}$ ) size of the  $\text{CaCO}_3$  particles suggest they could be vaterite, which is a metastable form of calcium carbonate. In the presence of water, vaterite would normally convert to calcite, however, polymer coating may have prevented transformation of vaterite to thermodynamically more stable calcite. XRD analysis indicates nearly equal amounts of calcite and vaterite in the sample, which suggests some of the vaterite may have transformed into calcite.



**Figure 32:** Secondary and backscattered electron images of precipitated solids in sample no. 1



**Figure 33:** Secondary and backscattered electron images of precipitated solids in sample no. 2

#### *4.1.1.3 50% DFF Mixed with 50% UFB (Sample No. 3)*

The XRD result shows Sylvite (KCl) is a major component of the sample. Calcium carbonates (calcite and vaterite ( $\text{CaCO}_3$ )) are also present in trace quantity.

The images and EDS X-ray spectrum of the precipitated solids in this sample are shown in Figure 34. The sample is predominantly KCl with small amounts of spherical 2  $\mu\text{m}$  particles of calcite and vaterite. The KCl unlike in other samples shows various forms of well developed crystalline structure of the cubic system. The  $\text{CaCO}_3$  particles also appeared to be agglomerated with polymer and seem to have co-precipitated with KCl. The XRD analysis confirms the presence of KCl, vaterite and calcite in this sample.

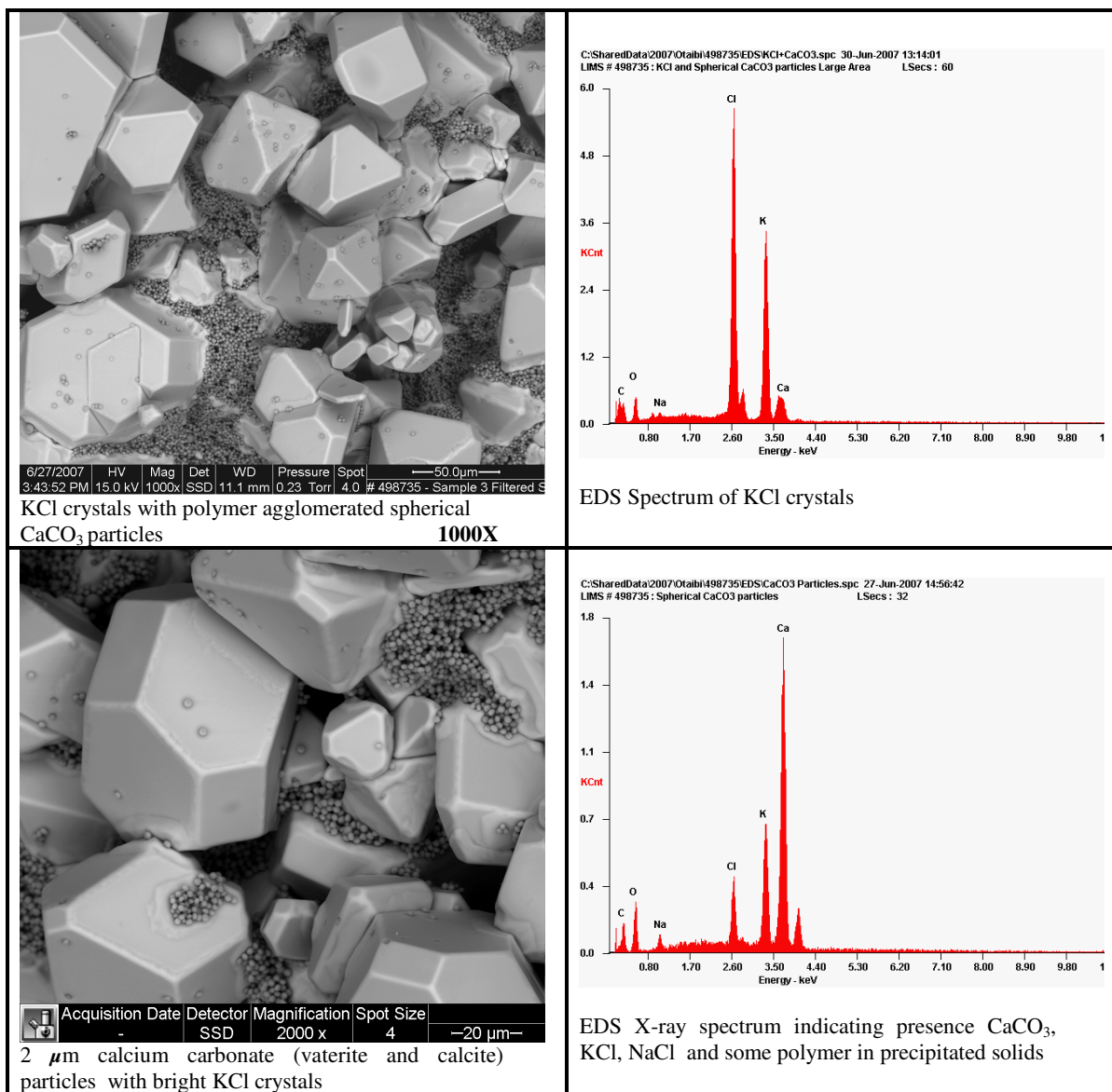
#### *4.1.2 UFB Mixed with PFB Solution at Room Temperature*

##### *4.1.2.1 10% UFB Mixed with 90% PFB*

Figure 35 shows backscattered electron images and EDS X-ray spectrum of the solids precipitated from mixing 10% UFB with 90% PFB at room temperature. The solids are mainly well crystallized 20-50  $\mu\text{m}$  KCl crystals that appear to be bonded by a thin porous layer of K-rich organic compound.

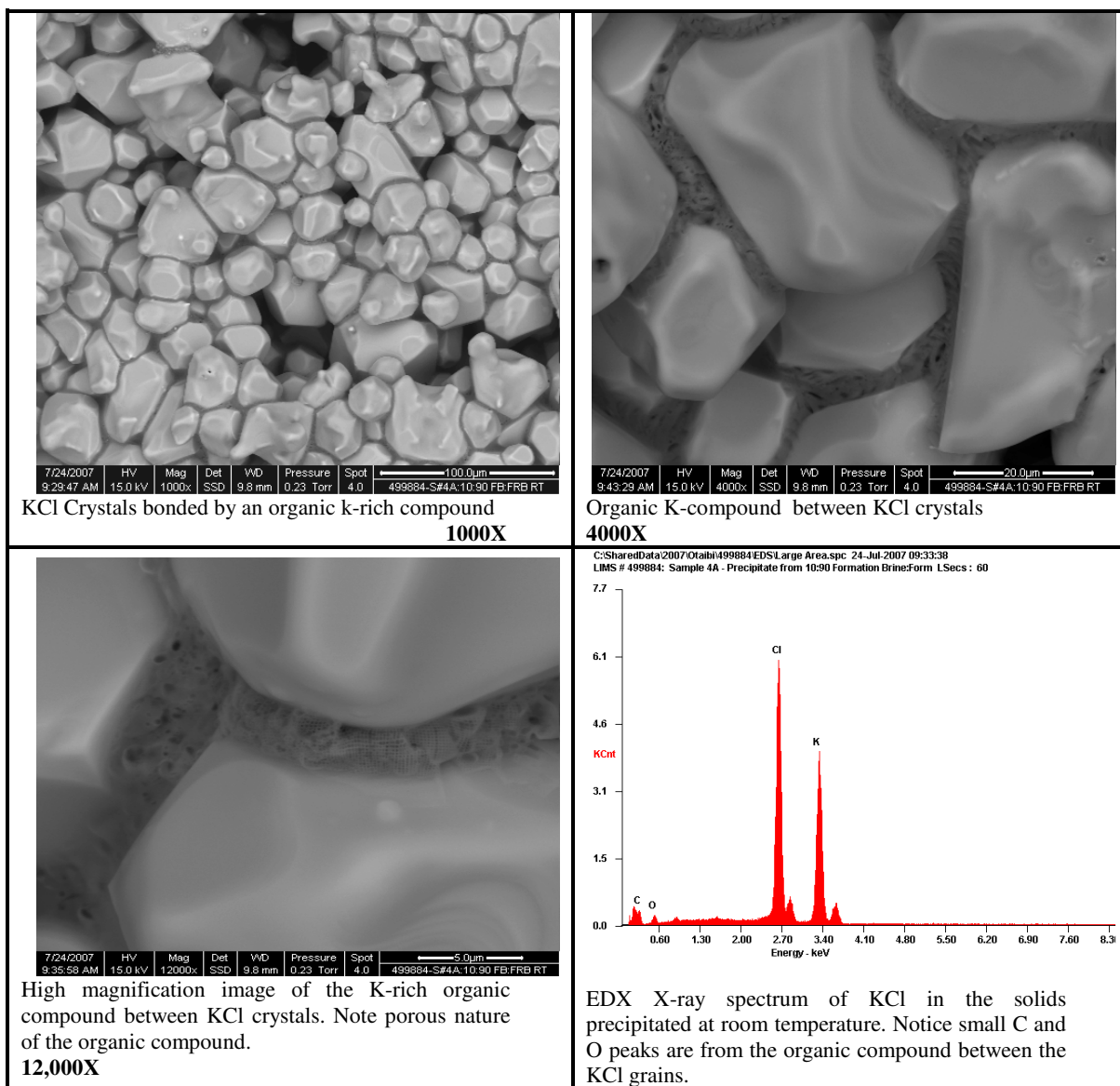
##### *4.1.2.2 30% UFB Mixed with 70% PFB*

Figure 36 shows images and EDS X-ray spectra of the solids precipitated from mixing 30% UFB with 70% PFB at room temperature. The solids are mainly 10-50  $\mu\text{m}$  crystals of Ca-rich compound that was identified by XRD as calcium formate. The solids also contain small amounts of well developed crystals of KCl.

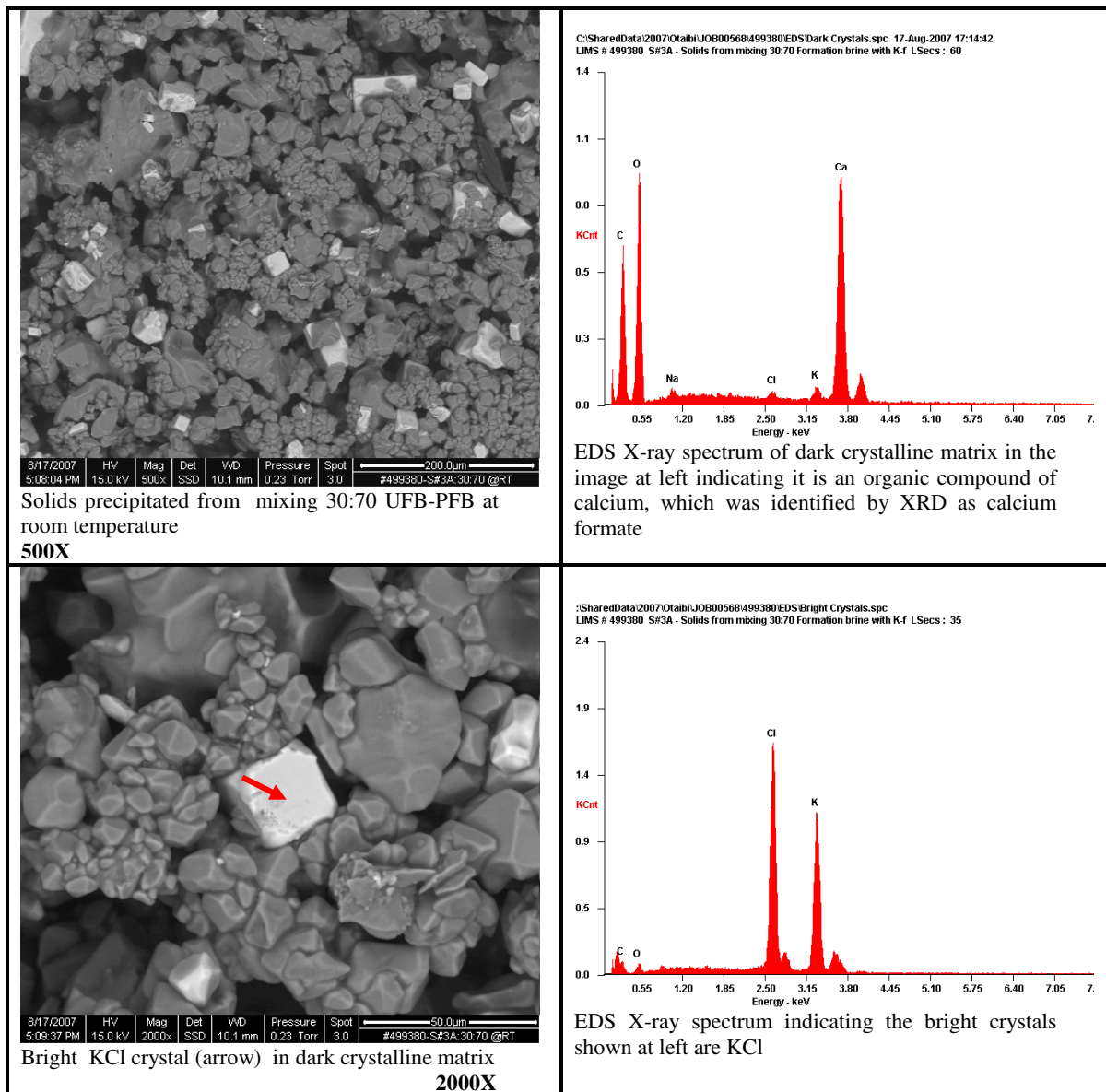


**Figure 34:** Secondary and backscattered electron images of precipitated solids in sample no. 3





**Figure 35:** Backscattered electron images and EDS X-ray spectrum of the solids precipitates at room temperature from mixing 10% UFB with 90% PFB



**Figure 36:** Backscattered electron images and EDS X-ray spectra of solids precipitated from mixing 30% UFB with 70% PFB at room temperature

#### *4.1.2.3 50% UFB Mixed with 50% PFB*

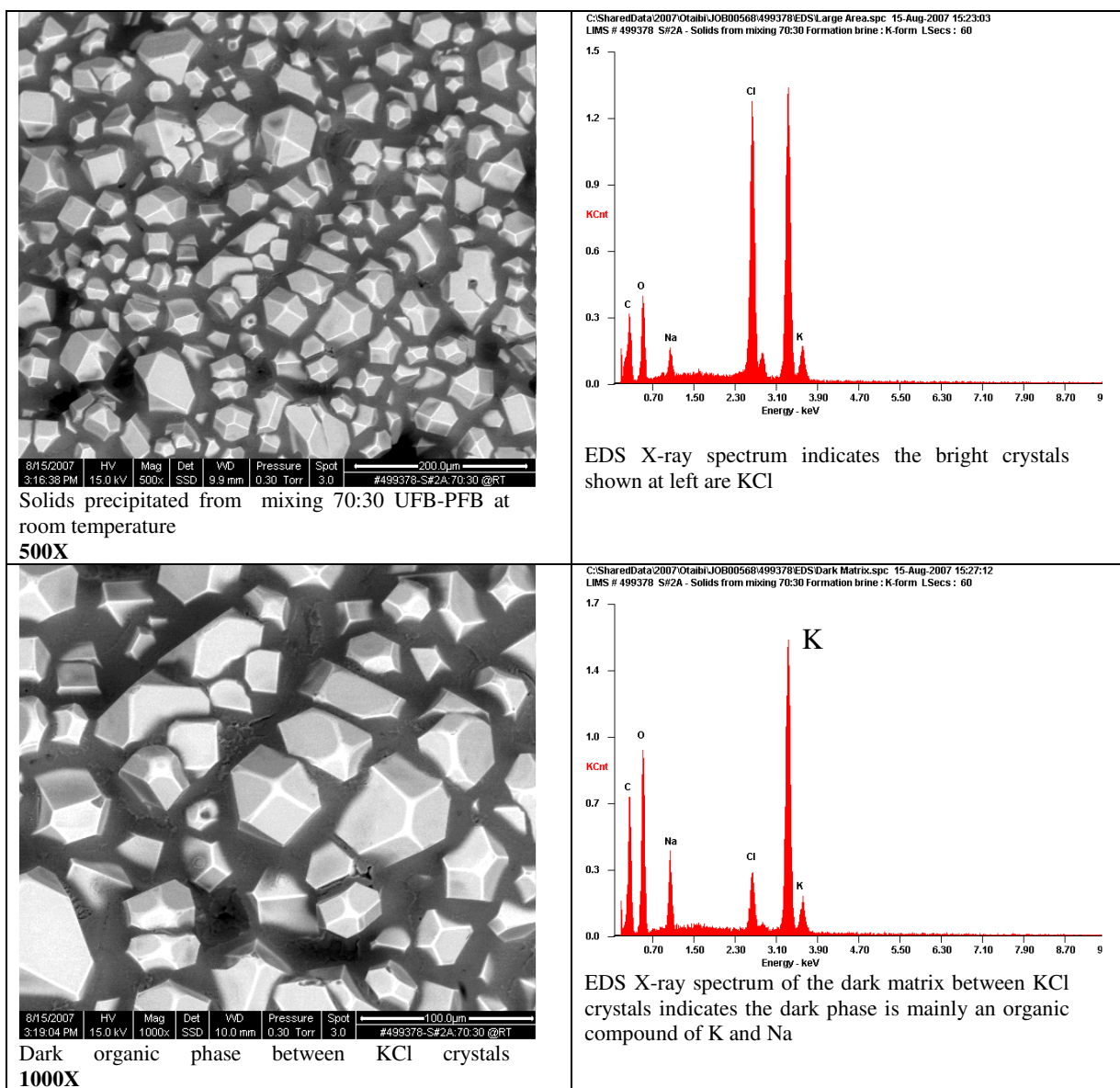
The solids precipitates that resulted from mixing 50% UFB with 50% PFB at room temperature were analyzed by using only XRD technique. The result shows the solids mainly consist of KCl.

#### *4.1.2.4 70% UFB Mixed with 30% PFB*

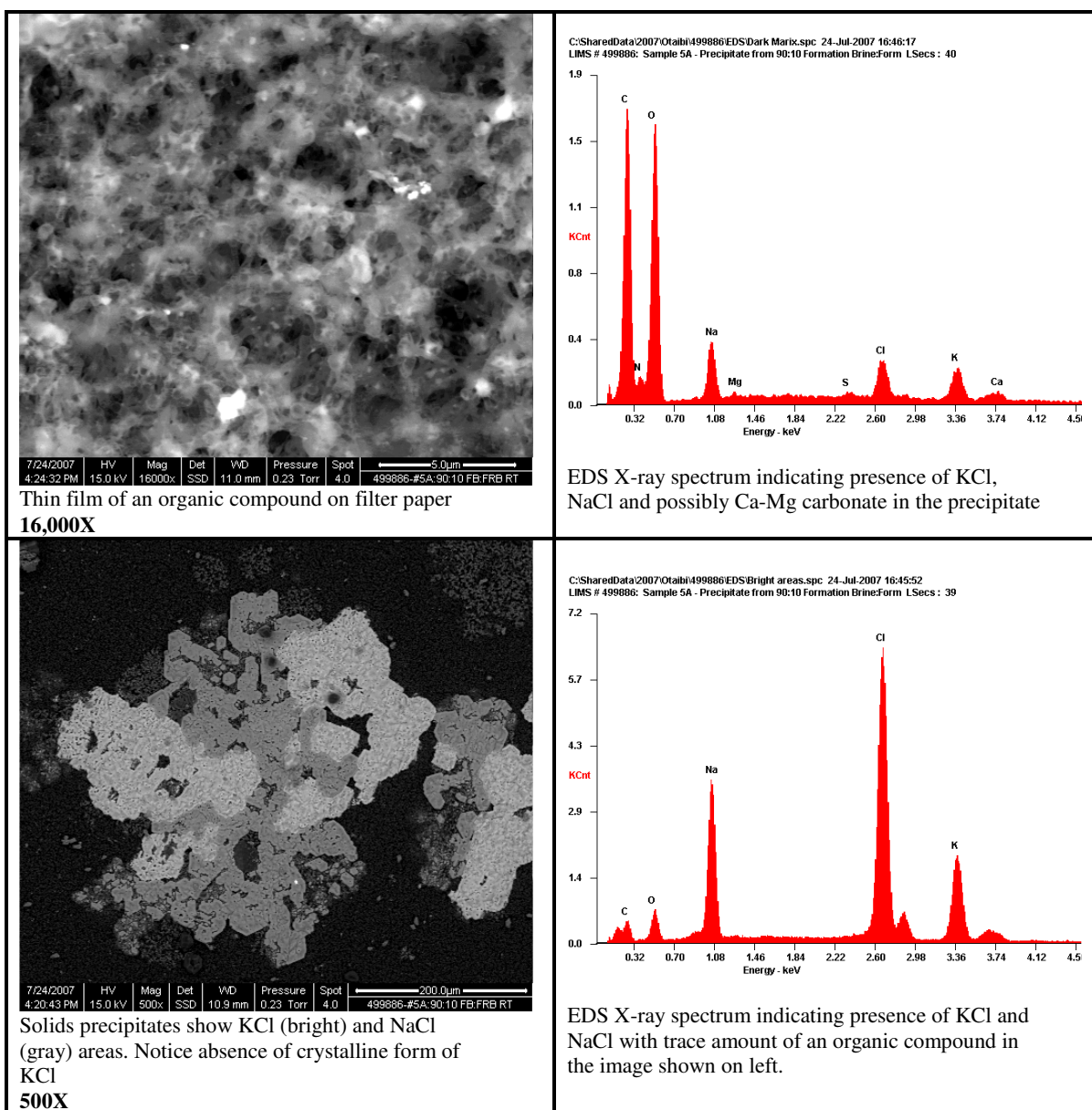
Figure 37 shows images and EDS X-ray spectrum of the solids precipitated from mixing 70% UFB with 30% PFB at room temperature. This sample also shows well crystallized KCl grains in a dark matrix. The EDS spectra of the matrix suggests it is an organic compound of K and Na, possibly K-formate and Na-Formate that were added to the formate brine.

#### *4.1.2.5 90% UFB Mixed with 10% PFB*

The EDS X-ray spectrum and the images of the solids precipitated from mixing 90% UFB with 10% PFB at room temperature are shown in Figure 38. The solids are mainly KCl and NaCl and what appears to be a very thin film of beam sensitive organic compound on filter paper. The elemental composition of the thin film could not be determined due to interference from the support filter paper. KCl and NaCl in this sample do not show crystalline form but appear as evaporated patches of brine. The EDS analysis also indicates presence of trace amount of Ca-Mg-Carbonate in the solids.



**Figure 37:** Backscattered electron images and EDS X-ray spectra of solids precipitated from mixing 70% UFB with 30% PFB at room temperature



**Figure 38:** Backscattered electron images and EDS X-ray spectrum of the solids precipitates at room temperature from mixing 90% UFB with 10% PFB

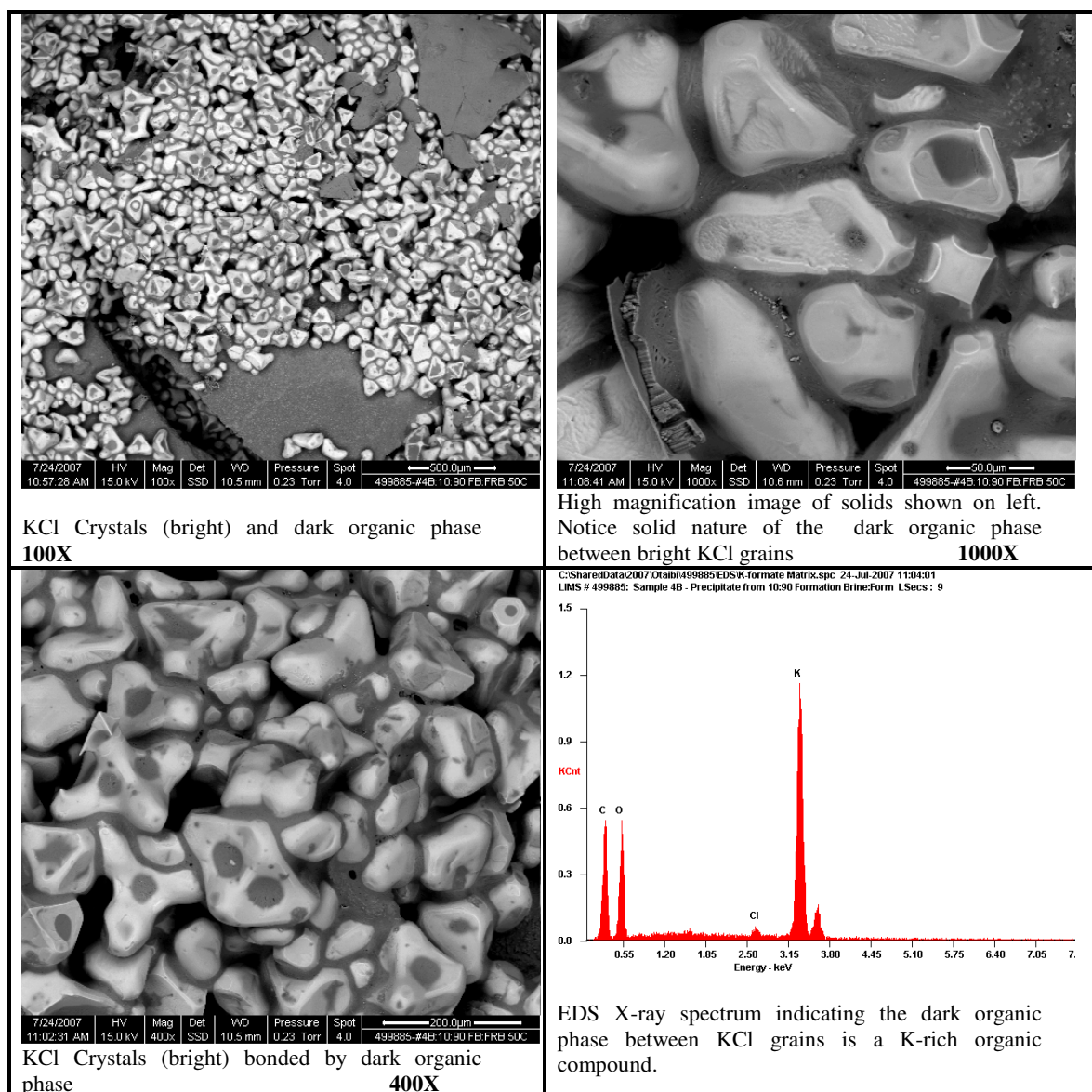
### *4.1.3 UFB Mixed with PFB Solution at High Temperature*

#### *4.1.3.1 10% UFB Mixed with 90% PFB*

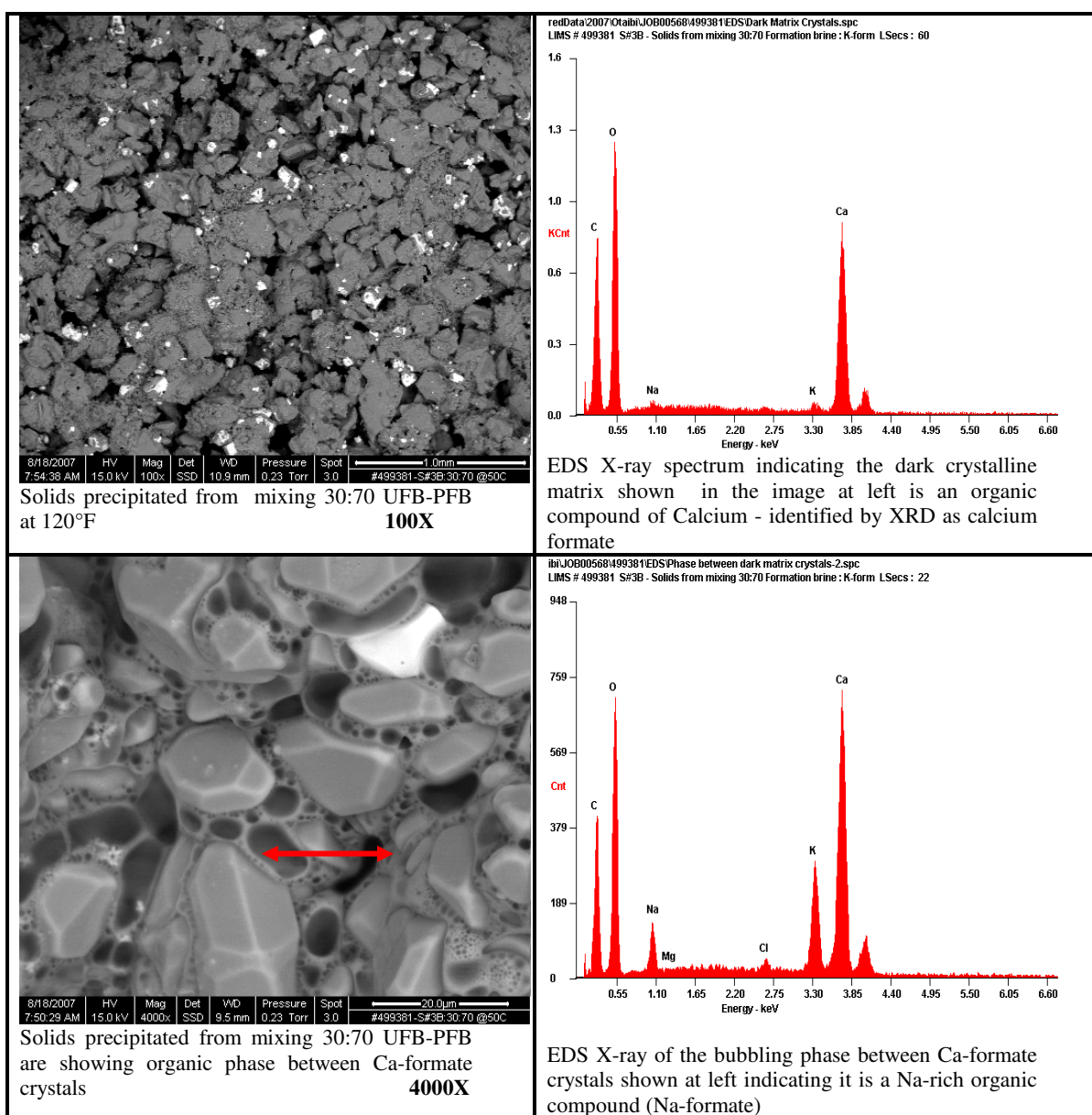
Figure 39 shows the images and EDS X-ray spectrum of the solids precipitated from mixing 10% UFB with 90% PFB at an average temperature of 120°F. As in the room temperature sample, this sample also shows well crystallized KCl grains bonded by K-rich organic compound. However, the organic compound because of the higher temperature appears to have become more dense and compact and also shows sheet-like morphology.

#### *4.1.3.2 30% UFB Mixed with 70% PFB*

Figure 40 shows images and EDS X-ray spectra of the solids precipitated from mixing 30% UFB with 70% PFB at average temperature of 120°F. The main solids as in the room temperature sample are also 10-50  $\mu\text{m}$  crystals of Ca-rich compound identified as calcium formate by XRD, and a few crystals of KCl. However, unlike the room temperature sample, this sample shows a Na-rich compound between Ca-formate grains. This Na-rich compound is highly unstable under the beam and appears to be Na-formate.



**Figure 39:** Backscattered electron images and EDS X-ray spectrum of the solids precipitates from mixing 10% UFB with 90% PFB



**Figure 40:** Backscattered electron images and EDS X-ray spectra of solids precipitated from mixing 30/70 UFB-PFB at 120°F

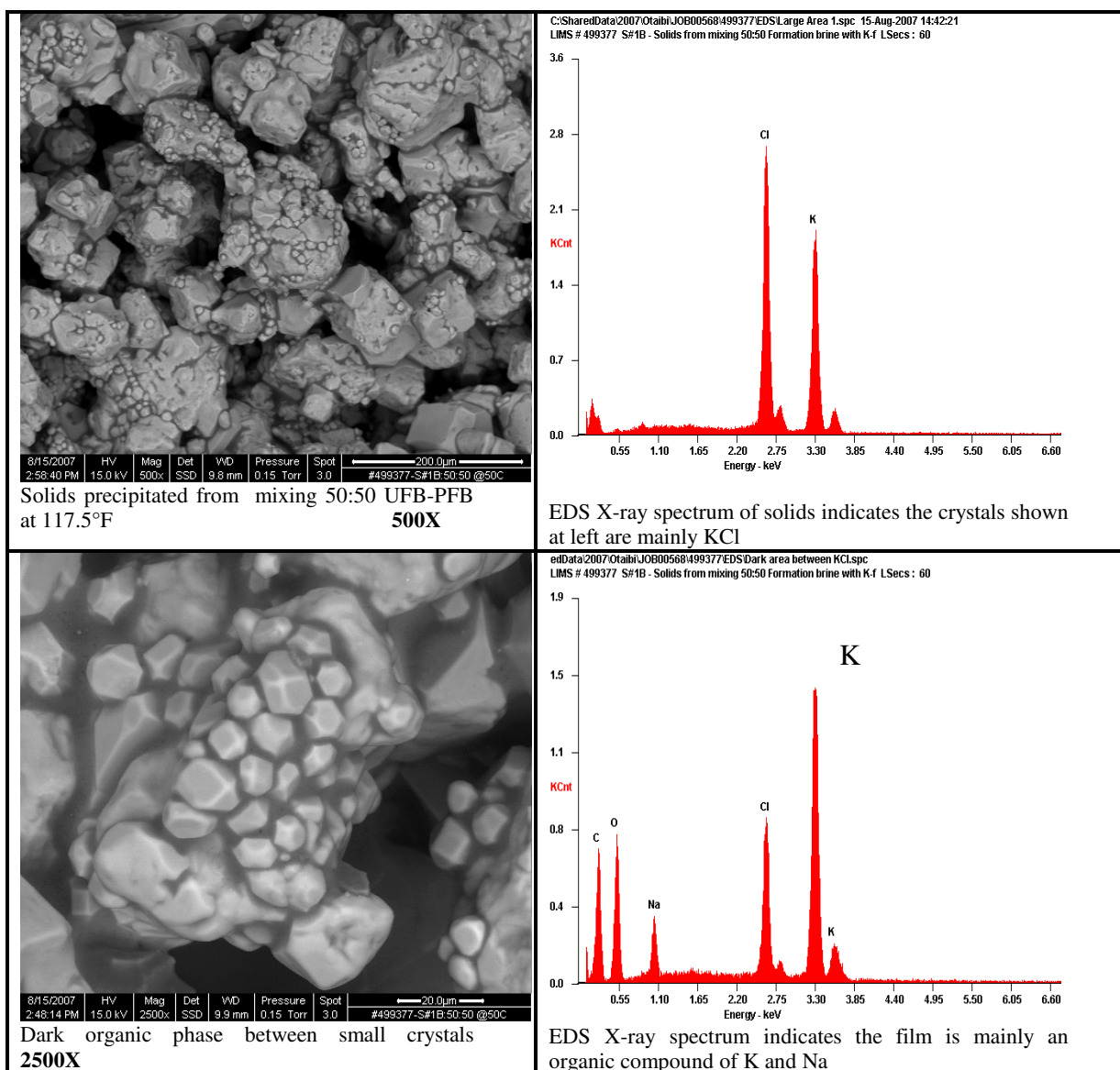


#### *4.1.3.3 50% UFB Mixed with 50% PFB*

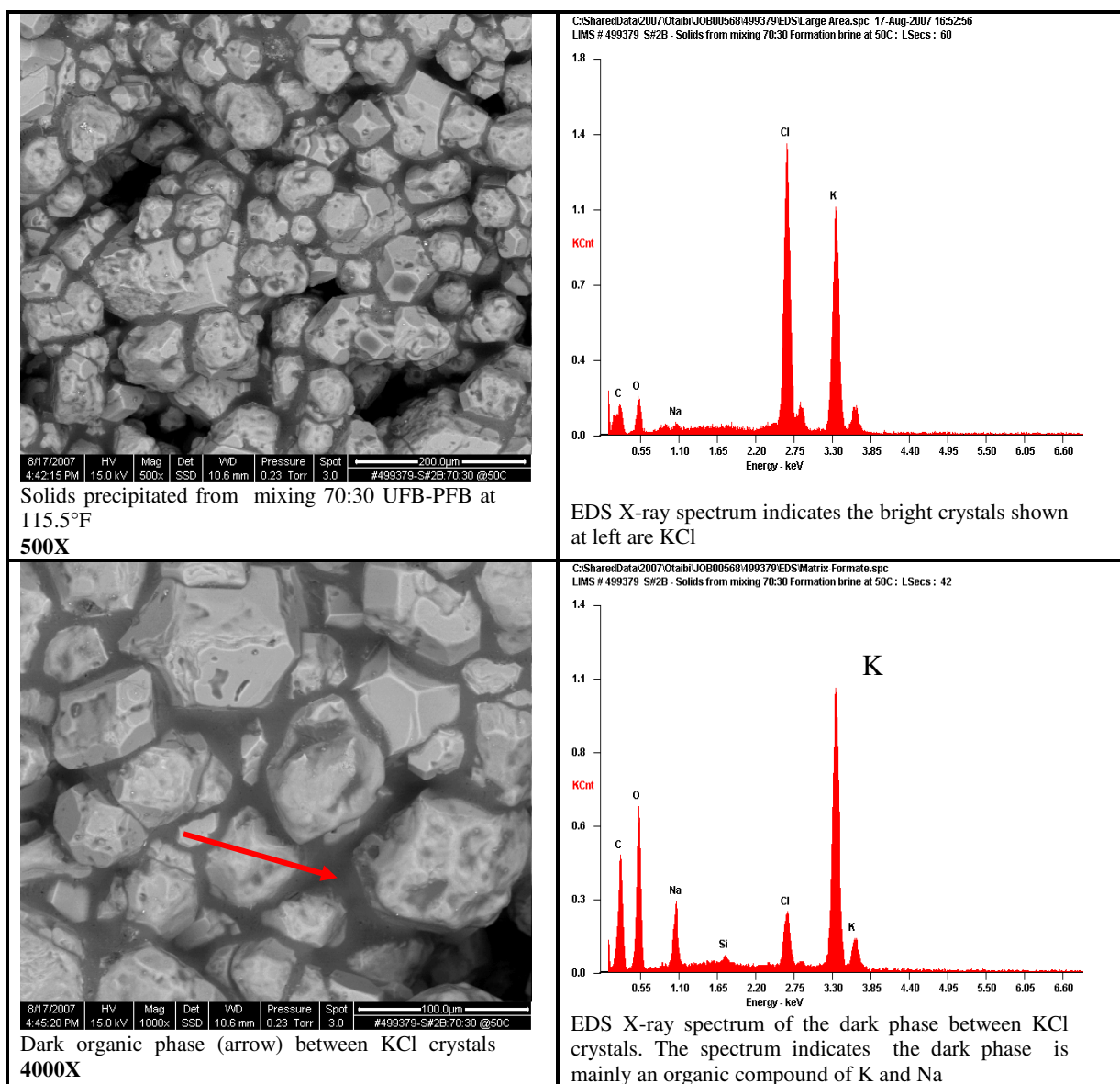
Figure 41 shows backscattered electron images and EDS X-ray spectrum of the solids precipitated from mixing 50% UFB with 50% PFB at an average temperature of 117.5°F. The solids are mainly well crystallized grains of KCl in the 50-100  $\mu\text{m}$  size range and some smaller crystals that appear to be bonded to larger crystals by a thin film of K and Na-rich organic compounds.

#### *4.1.3.4 70% UFB Mixed with 30% PFB*

The images and EDS X-ray spectra of solids precipitated from mixing 70% UFB with 30% PFB at 115.5°F are shown in Figure 42. Except for minor differences in the smoothness of the faces of precipitated crystals, the results show that there is virtually no difference in the composition of the solids between 115.5°F and room temperature samples. The precipitated solids at 115.5°F are also KCl crystals that are dispersed in an organic matrix. The EDS spectrum of the matrix suggests it could be K-formate with some Na-formate.



**Figure 41:** Backscattered electron images and EDS X-ray spectra of solids precipitated from mixing 50:50 UFB and PFB at 117.5°F



**Figure 42:** Backscattered electron images and EDS X-ray spectra of solids precipitated from mixing 70:30 UFB-PFB at 115.5°F

#### *4.1.3.5 90% UFB Mixed with 10% PFB*

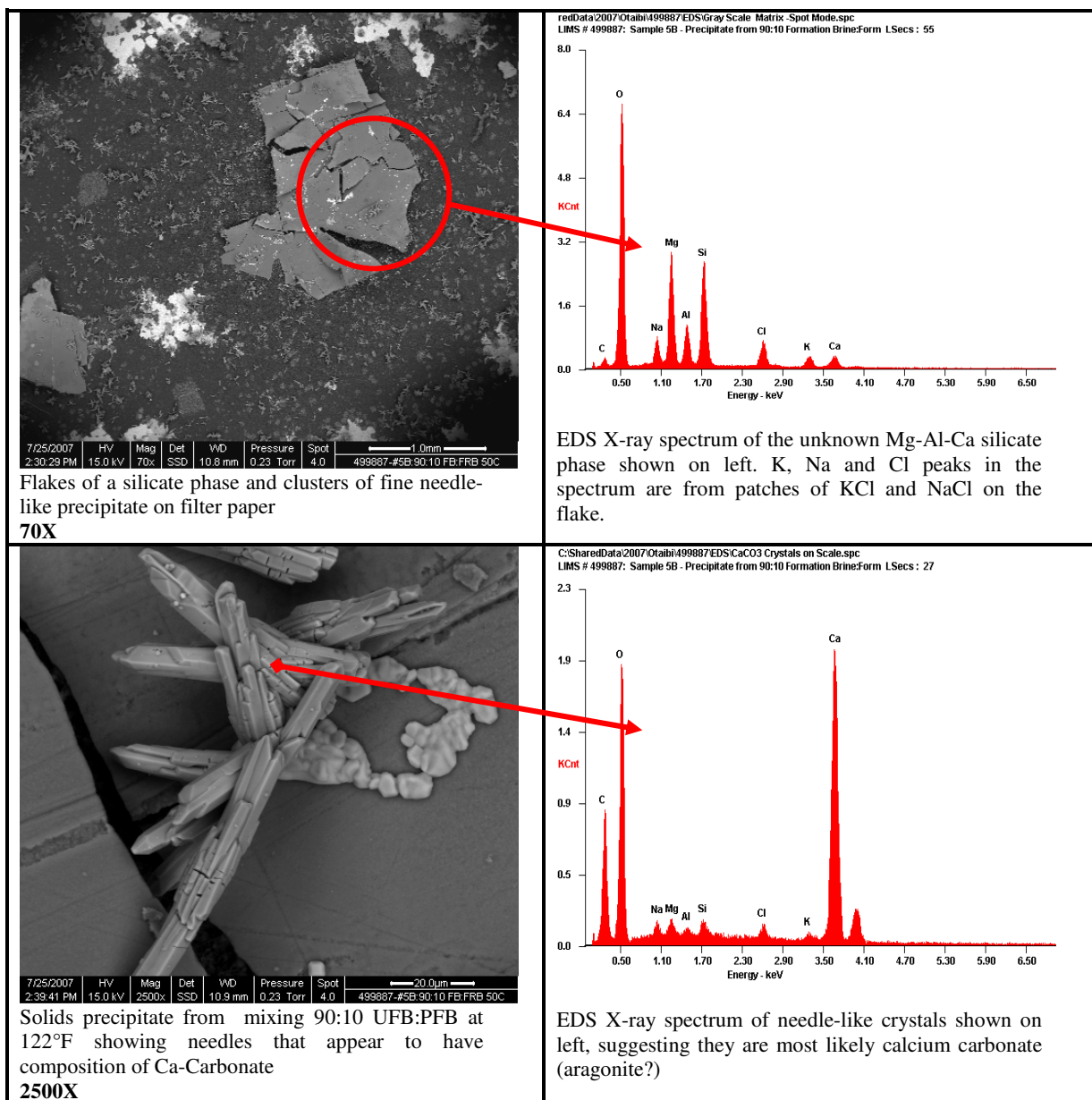
The images and EDS X-ray spectrum and the images of the solids precipitated from mixing 90% UFB with 10% PFB at 122°F are shown in Figure 43. The solids are mainly flakes of an unknown silicate phase that contains Mg, Al, and Ca besides Si and O, needles of calcium carbonate, possibly aragonite and patches of KCl and NaCl. Trace amounts of an organic compound also appear to be present as a thin film on filter paper. The presence of a significant amount of the silicate phase in this sample was unusual as it was not observed in the corresponding room temperature sample.

#### *4.1.4 Organic Acids Mixed with PFB Solution*

##### *4.1.4.1 10 wt% Citric Acid Mixed with PFB at Room Temperature*

As discussed in the fluid compatibility part, citric acid was forming a solid ball at room temperature during the mixing with PFB. The ball was first separated from the solution, and then the solution was filtered through 1.2  $\mu\text{m}$  filter paper. All the solids were analyzed by utilizing both XRD and ESEM techniques.

The XRD result shows the ball solid mainly consists of potassium hydrogen citrate ( $\text{C}_6\text{H}_7\text{KO}_7$ ) and potassium formate ( $\text{CHKO}_2$ ). Background hump in diffractogram suggest presence of amorphous components in the sample. There are a few minor peaks are not identified in the sample.

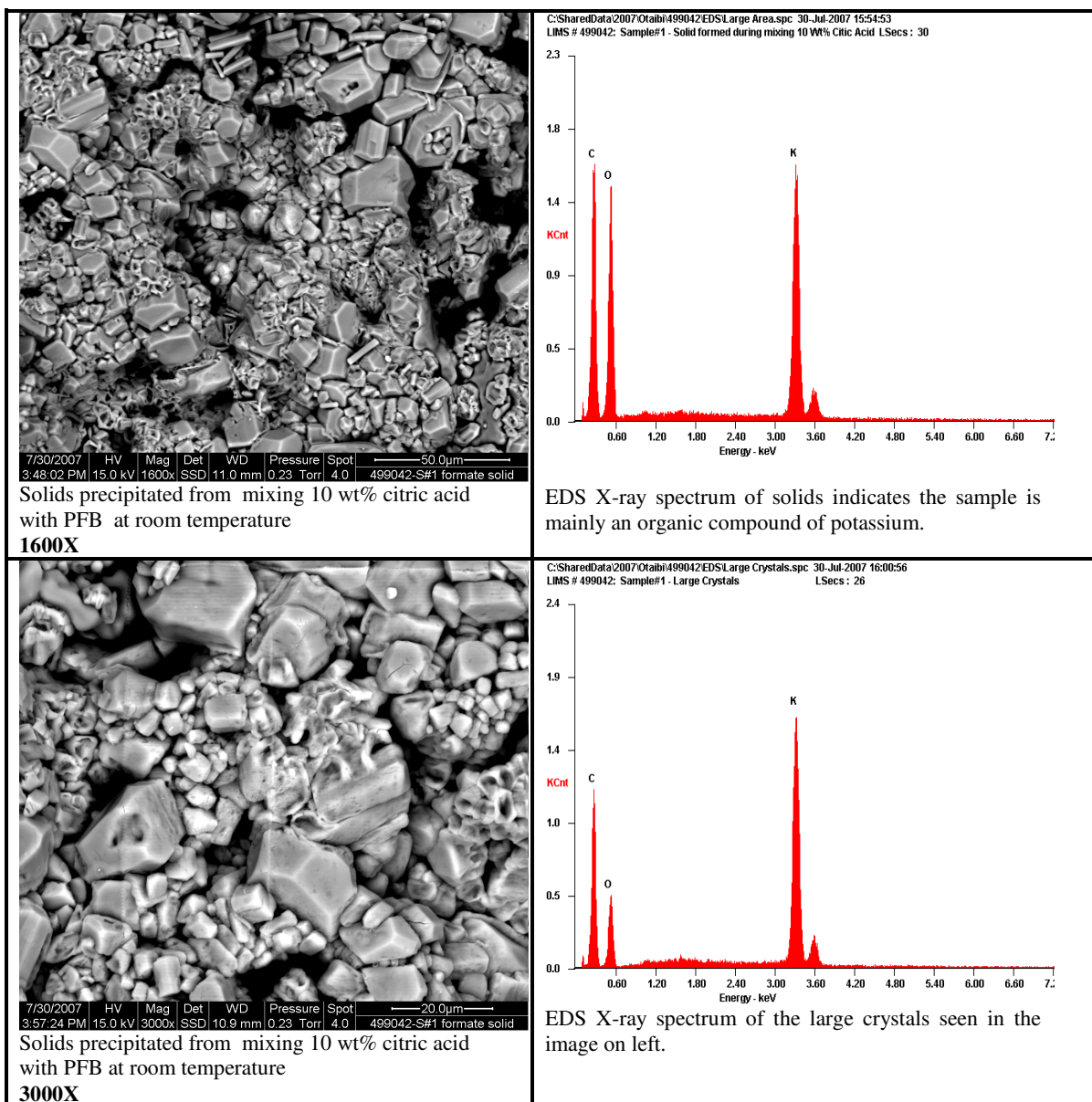


**Figure 43:** Backscattered electron images and EDS X-ray spectrum of the solids precipitates at 122°F from mixing 90% UFB with 10% PFB

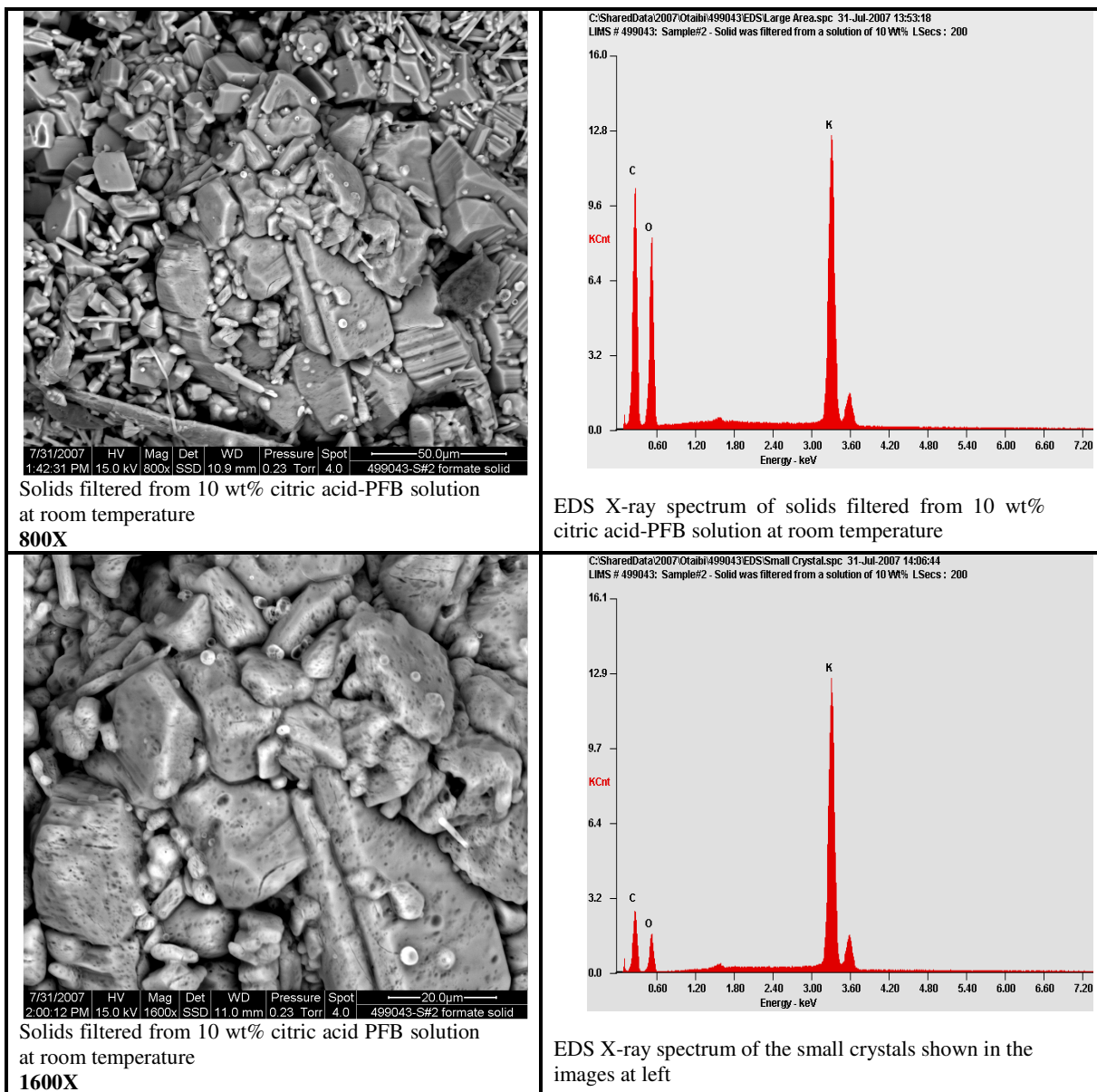
Figure 44 shows backscattered electron images and EDS X-ray spectra of the solids precipitated during mixing 10 wt% citric acid with PFB at room temperature. The solids in this sample show well defined crystalline form and a bimodal size distribution. The EDS analysis of the large (~20  $\mu\text{m}$ ) and small (<5  $\mu\text{m}$ ) crystals indicate that they both contain K, O and C, which suggests they are most likely organic compounds of potassium. The XRD analysis indicates the solids are a mixture of K-formate and K-citrate. This indicates that the two types (sizes) of crystals observed in the ESEM probably correspond to two different compounds, namely K-citrate and K-formate. However, because of the qualitative nature of light element analysis in the ESEM it was not possible to determine which size crystals correspond to which compound.

The filtered solid on the paper contains minor quantities of potassium hydrogen citrate ( $\text{C}_6\text{H}_7\text{KO}_7$ ) and potassium formate ( $\text{CHKO}_2$ ). Potassium chlorate ( $\text{KClO}_3$ ) might also be present in the sample. Background hump in diffractogram suggest major portion of the sample consists of amorphous components. No precipitates were noticed when the mixture was prepared at high temperature.

Figure 45 shows the images and EDS X-ray spectra of the solids filtered from a mixture of 10 wt% citric acid and PFB at room temperature. As in the previous sample, this sample shows well crystallized grains and their EDS spectra show K, O and C, indicating the solids are mainly organic compounds of potassium. The XRD analysis indicates K-formate and K-citrate are the main two phases in the sample, which suggests there is no noticeable difference between the two samples.



**Figure 44:** Backscattered electron images and EDS X-ray spectra of solids precipitated during mixing of 10 wt% citric acid with PFB at room temperature



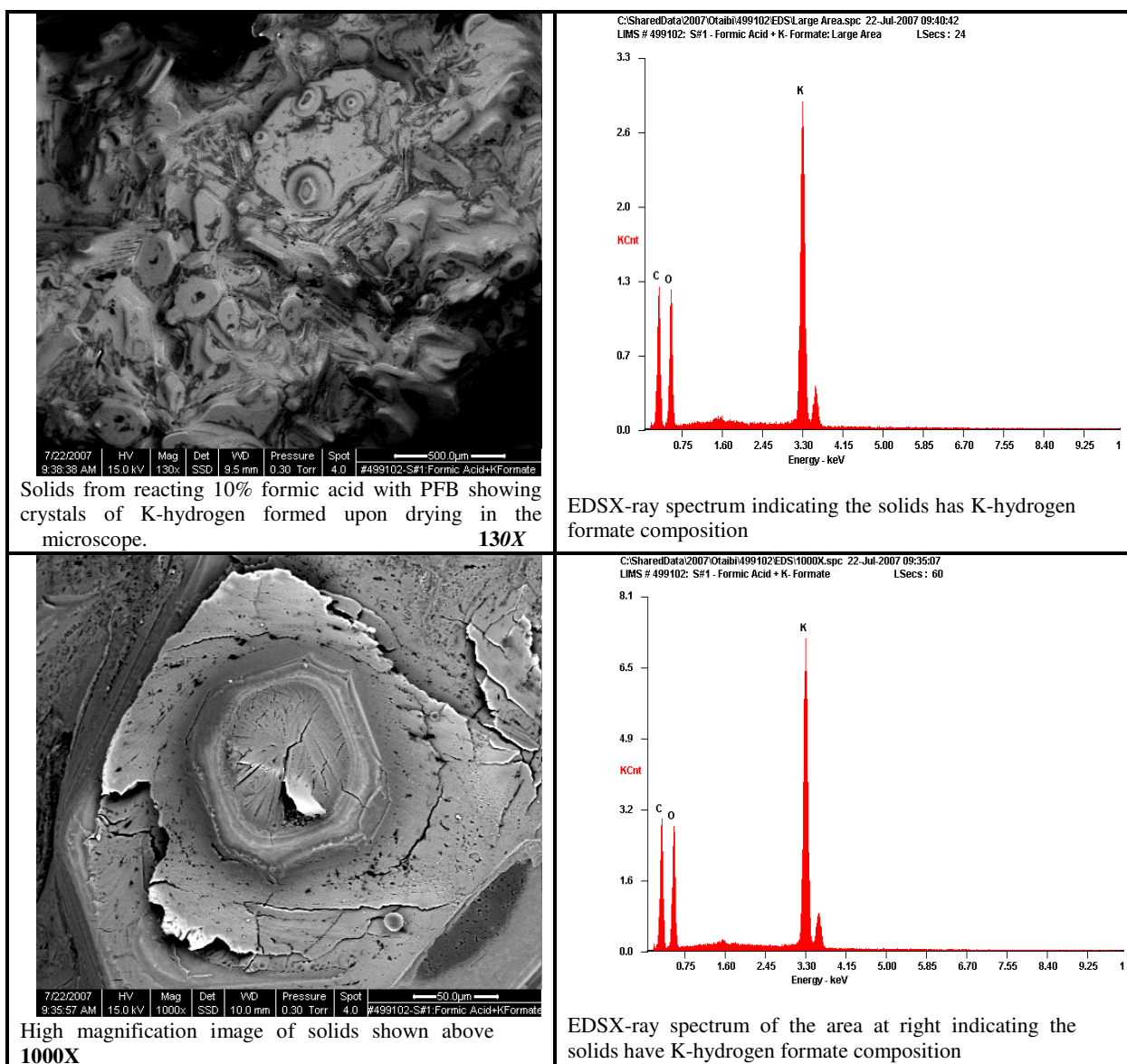
**Figure 45:** Backscattered electron images and EDS X-ray spectra of solids filtered from 10 wt% citric acid-PFB solution at room temperature



#### 4.1.4.2 10 wt% Formic Acid Mixed with PFB at Room Temperature

Formic acid shows incompatibility with PFB solution at room temperature. At high temperatures, the mixture was compatible but when temperature reduced to the room temperature, it became a gel. The precipitates were analyzed by XRD first, and the result shows it is potassium hydrogen formate compound ( $C_2H_3KO_4$ ).

Figure 46 shows images and EDS X-ray spectra of the solid formed from reaction of 10% formic acid with PFB. The results indicate the wet solid which crystallized under high vacuum in the microscope has the composition of K-hydrogen formate. Such wet corrosive solids are not suitable for examination in the ESEM since during initial pump down cycle the samples dry out and the corrosive gases released could easily damage to the delicate components of the microscope. In addition, the samples can also show some drying artifacts that might be not present in the original sample.



**Figure 46:** Backscattered electron images and EDS Spectra of solids formed from 10% formic acid reaction with PFB at room temperature

## 4.2. Analysis of Dynamic HPHT Filter Press Cell Experiments

### 4.2.1 *Drilling Fluid Experiment*

Field drilling fluid samples were used in all lab experiments and that sample was collected from a rig site while drilling a gas well. The drilling fluid formulation and properties are shown in Table 2. 1.2  $\mu\text{m}$  filter paper was utilized at the beginning to filter the drilling fluid solids at room temperature. It was difficult to filter all solids at room temperature. Therefore, the filter paper was replaced twice. A static HPHT filter press was employed to make sure most of the solids are filtered. A 3- $\mu\text{m}$  ceramic disk was used, instead of the filter paper, at temperature and pressure of 300°F and 200 psia, respectively. Then, the solids were dried in the oven at a temperature of 194°F for 3 hours. Solid samples presented on both filter papers and ceramic disk were analyzed by using XRD technique. Table 6 shows XRD result for the solids, they were mainly calcite and dolomite. Dolomite concentration was more than 15 wt% and the source of that were the impurities of the calcium carbonate particles, which used in drilling fluid. The source of the barite was the drilling fluid that was used to drill the upper hole section. A minor quantity of quartz was present in the filter cake. Core plugs samples from an offset well were crushed and sent to XRD for analysis. The results showed 97 wt% of the solids was quartz ( $\text{SiO}_2$ ) and the rest 3 wt% was kaolinite ( $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_5$ ).

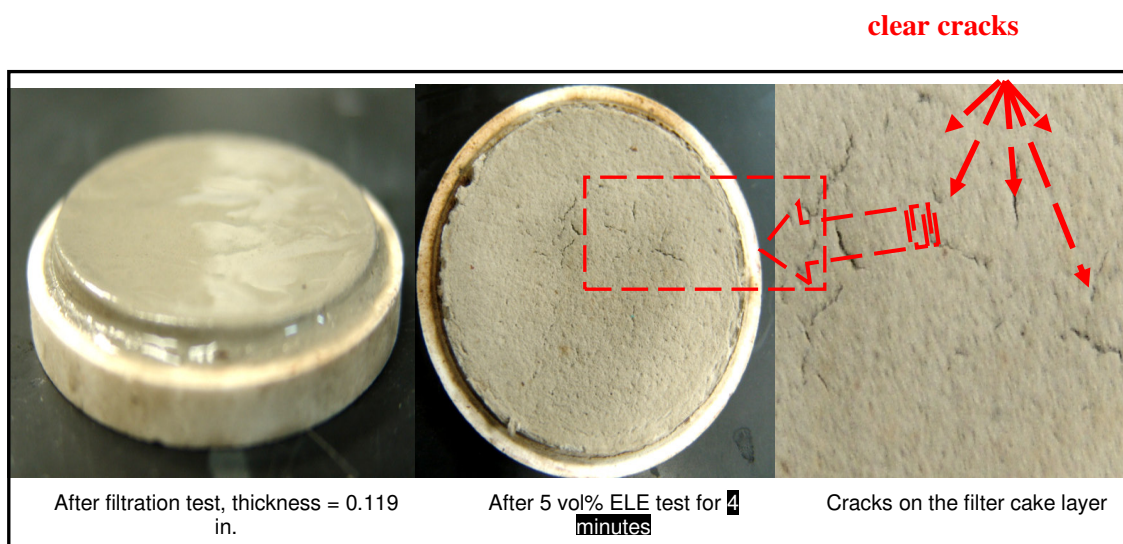
**Table 6:** Solids filtered from field drill-in fluid sample

Compound	Formula	Filter Paper Size (1.2 $\mu\text{m}$ )			Ceramic Disk (3 $\mu\text{m}$ )
		Filtration conditions at room temperature			at 250°F and 200 psi
		Filter paper # 1	Filter paper # 2	Filter paper # 3	
		Concentration, wt%			
Calcite	CaCO <sub>3</sub>	72	66	72	71
Dolomite	CaMg(CO <sub>3</sub> ) <sub>2</sub>	17	20	16	17
Barite	BaSO <sub>4</sub>	6	8	6	7
Quartz	SiO <sub>2</sub>	5	6	6	5

#### 4.2.2 5 vol% Ethyl Lactate Ester (ELE)

Potassium chloride brine was used to measure the base flow rate, through 3- $\mu\text{m}$  undamaged disk, at a temperature of 303°F and a pressure of 50 psia. Then, formate drilling fluid was loaded in the filter press cell at the same temperature and 200 psia overbalanced pressure. The motor shaft rotated at 100 rpm to simulate the dynamic condition while drilling operation. The cumulative filtrate volume was collected as a function of time. The cell was disconnected and the filter cake thickness was measured, Figure 47. A 5 vol.% ethyl lactate ester (ELE) solution was prepared in KCl brine solution as shown in Table 7. The ester solution, 200 ml, was soaked for 30 minutes in the cell after the reservoir temperature was reached. Then, the stem valve at the bottom of the cell cap was fully opened. The objective behind this step was to determine the breakthrough time after dissolving the filter cake layer. The breakthrough time of the reaction was expected to be between 3 to 4 hours since it is a weak acid. Most of the ester solution passed through the valve within 4 minutes. The solution channeled through the weakest point in the ceramic disk. Small cracks were created on the face of the disk

as shown on Figure 47. The flow rate of KCl brine through the ceramic disk was measured before the damage and after soaking the disk with ester solution. Removing efficiency can be calculated based on the initial and final brine flow rate. The soaking time for the ester solution was not enough to clean the filter cake. The remaining solids on the disk were sent to XRD, EDXRF and ESEM for further analysis. The result showed 58 wt% calcite ( $\text{CaCO}_3$ ), 15 wt% sylvite ( $\text{KCl}$ ), 10 wt% dolomite ( $\text{CaMg}(\text{CO}_3)_2$ ) and 10 wt% barite ( $\text{BaSO}_4$ ). The EDXRF analysis is given in Table 8.



**Figure 47:** Filter cake after filtration test and 5 vol% ethyl lactate ester (ELE), temperature = 303°F and pressure = 200 psi.

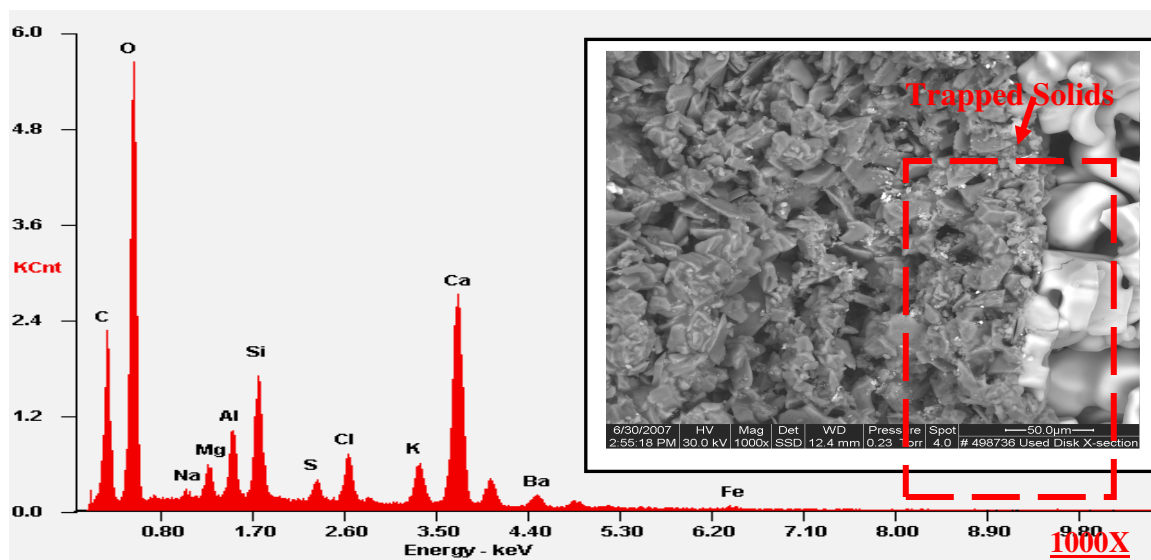
**Table 7:** Formula of ethyl lactate ester cleaning solution

Component	Quantity for 5 and 10 vol% solution		Units
Water	94.5	89.5	cm <sup>3</sup>
KCl	5	5	g
Acid Precursor	5	10	cm <sup>3</sup>
Nonionic Surfactant	0.2	0.2	cm <sup>3</sup>
Corrosion inhibitor	0.3	0.3	cm <sup>3</sup>
pH @ 79°F	3.59	3.00	—
Density @ 79°F	64.93	65.05	lb/ft <sup>3</sup>

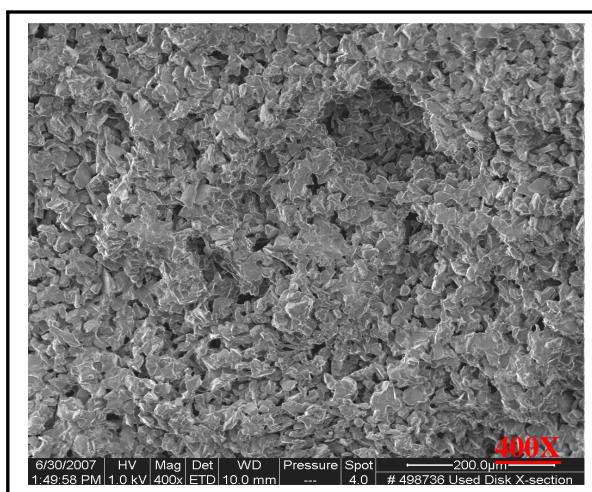
**Table 8:** EDXRF result for solids on the surface of the ceramic disk after 5 vol% ethyl lactate ester

Element	Ca	Ba	K	Cl	Si	Na	Mg	S	Al	Fe	Sr	Mn	P	Zn
Concentration	23	8.3	8.2	7.3	4.7	1.8	1.6	0.9	0.6	0.4	700	600	<500	<500
Unit	wt. %										mg/kg			

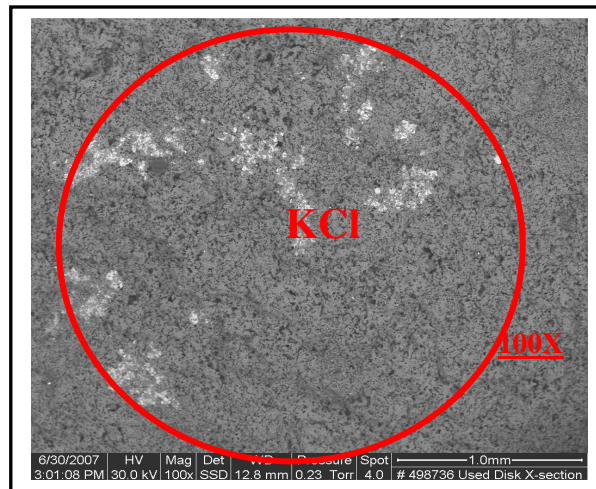
The ESEM results confirmed the presence of the previous compounds in XRD test in addition to 7 wt% quartz (SiO<sub>2</sub>). The disk was broken to small pieces to do a cross section analysis. The EDS X-ray spectrum of the solids below the disk surface is shown in Figure 48 suggests presence of calcite, barite, sylvite and some silicates in the solids. The SEM image of the disk cross-section showed bright KCl layer on the surface and very fine particles trapped in the pores to about 50 μm below surface, Figures 49 and 50. The EDS X-ray spectrum of filter cake solids on the ceramic disk surface indicated presences of CaCO<sub>3</sub>, BaSO<sub>4</sub>, SiO<sub>2</sub> and possibly some clay particles, Figure 51. Trace amounts of heavy mineral zircon (zirconium silicate), and a few Fe-Cr particles were also observed in the disk just below the surface.



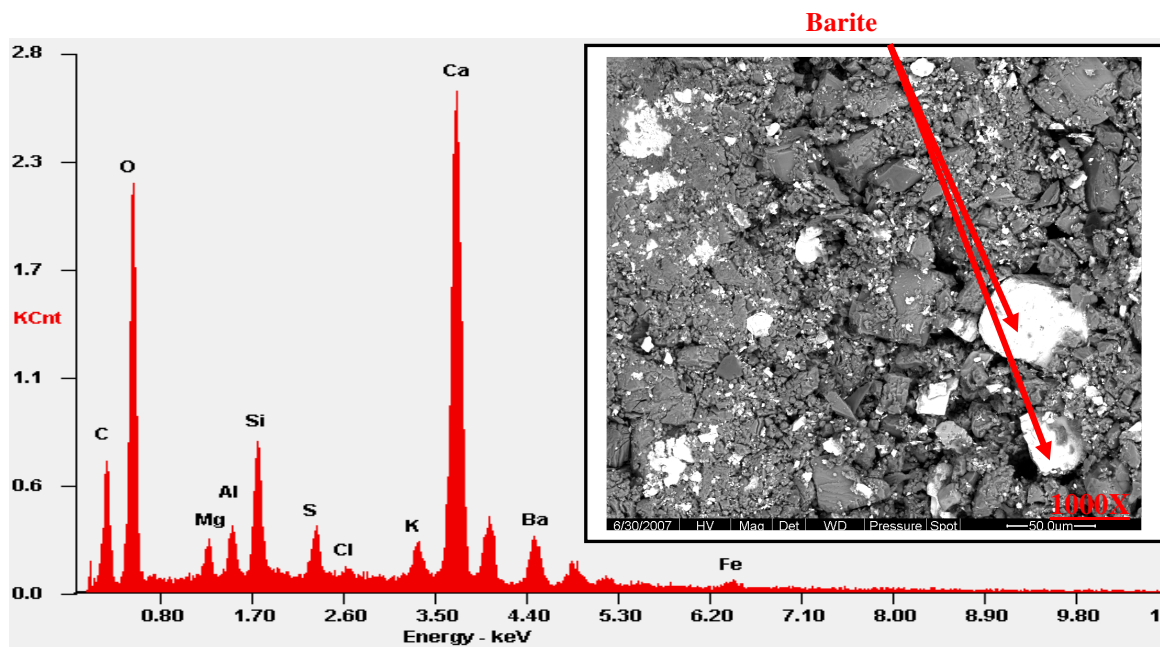
**Figure 48:** EDS X-ray spectrum of the disk cross-section after 5 vol% ethyl lactate ester



**Figure 49:** BSE image of the disk cross-section KCl patches in the interior of the disk



**Figure 50:** BSE image of the disk showing bright showing absence of solids in the interior of the disk

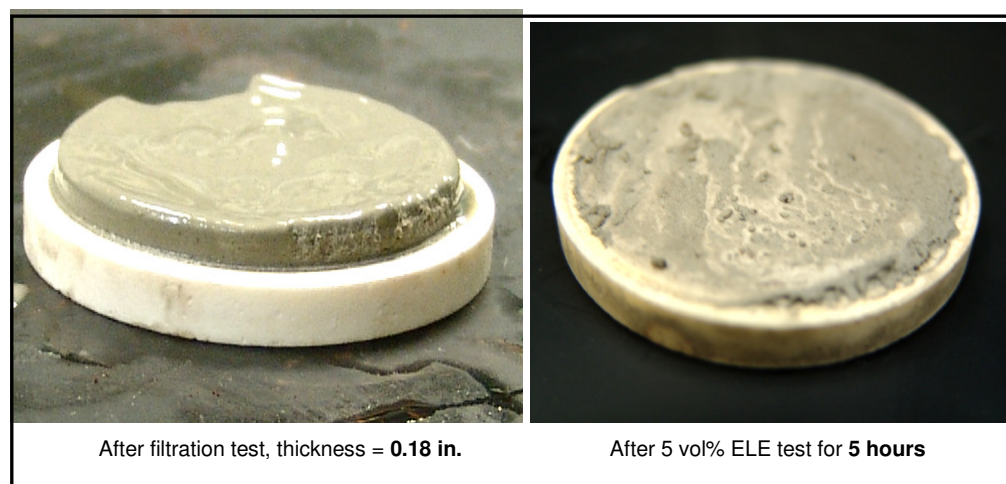


**Figure 51:** EDS X-ray spectrum of filter cake solids on ceramic disk



#### 4.2.3 5 vol% ELE for 5 hours

The second experiment was conducted by following the same procedure in the first test. The same drilling and cleaning fluids were used in this test and the only difference was the soaking time. The thickness of the filter cake was 0.18 in. after the filtration test step. A base flow rate was measured by using KCl fluid after the damage was created and the result was negligible. The 5 vol% lactate ester solution was left 5 hours for soaking, at a temperature range between 300 to 315°F. The generated lactic acid solution was able to dissolve most of the filter cake and the removing efficiency was above 80%, Figure 52.

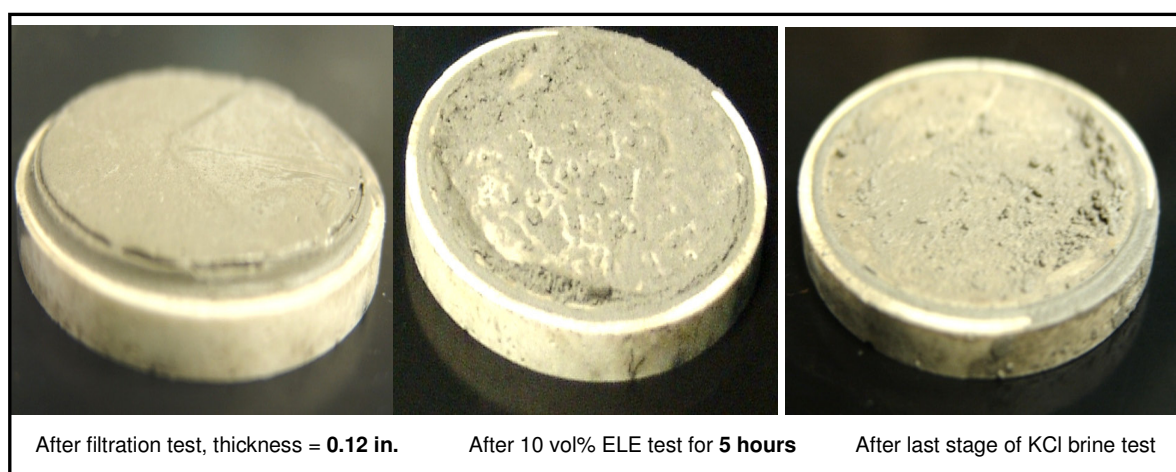


**Figure 52:** Filter cake after filtration test and 5 vol% ethyl lactate ester (ELE), temperature = 300-315°F and pressure = 200 psi.

#### 4.2.4 10 vol% ELE

In this experiment, the concentration of the ester solution was increased to be 10 vol% to dissolve more calcium carbonate particles. The cake thickness was 0.119 in. and the test temperature was 298°F. The cell was filled with KCl brine to measure the flow through

the damaged disk. The recorded brine volume through the cake was almost zero. The ELE solution was soaked for 5 hrs at temperature and pressure of 304°F and 200 psi, respectively. Increasing the concentration of the ester solution did significant improve the filter cake cleanup efficiency as shown in Figure 53.

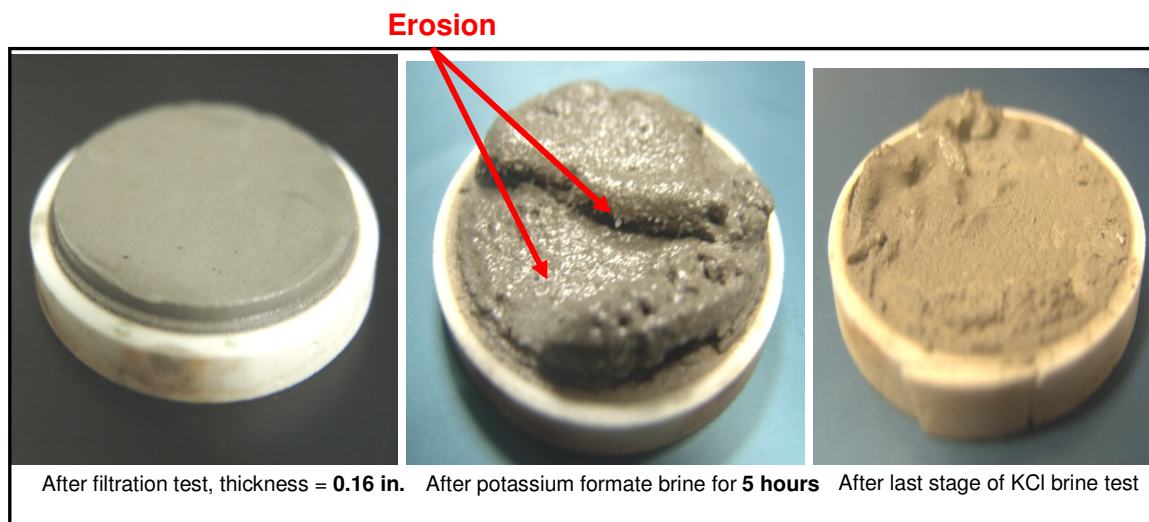


**Figure 53:** Filter cake after filtration test and 10 vol% ethyl lactate ester (ELE), temperature = 300°F and pressure = 200 psi.

#### 4.2.5 Solids-free Potassium Formate Brine

Solids-free potassium formate brine has been commonly used in the field to remove the filter cake damage at both static and dynamic conditions. The aim of this experiment was to examine the effect of formate brine in removing filter cake damage mechanically. The same procedure was followed, as done in previous experiments, and the cake thickness was found to be 0.16 in. The solids-free potassium formate brine was loaded in the cell and the rotating shaft was fixed at 100 rpm simulating field operation. The cell

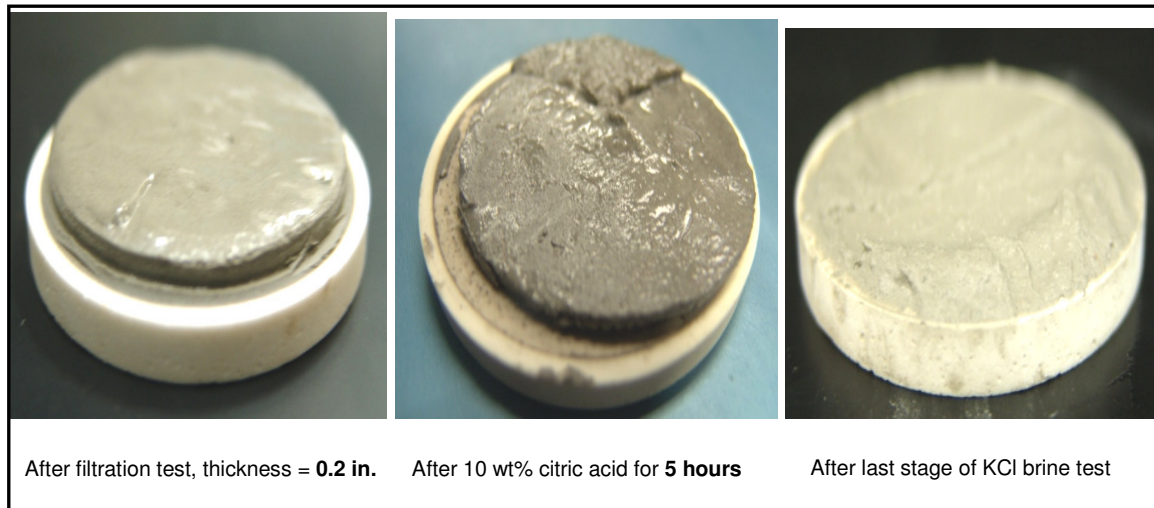
temperature was kept at 300°F and the pressure at 200 psi. The cell was rotating for 5 hours, and the formate brine solution was able to remove only a small portion of the external filter cake layer as shown in Figure 54.



**Figure 54:** Filter cake after filtration test and solids-free potassium formate brine, temperature = 300°F and pressure = 200 psi.

#### 4.2.6 10 wt% Citric Acid

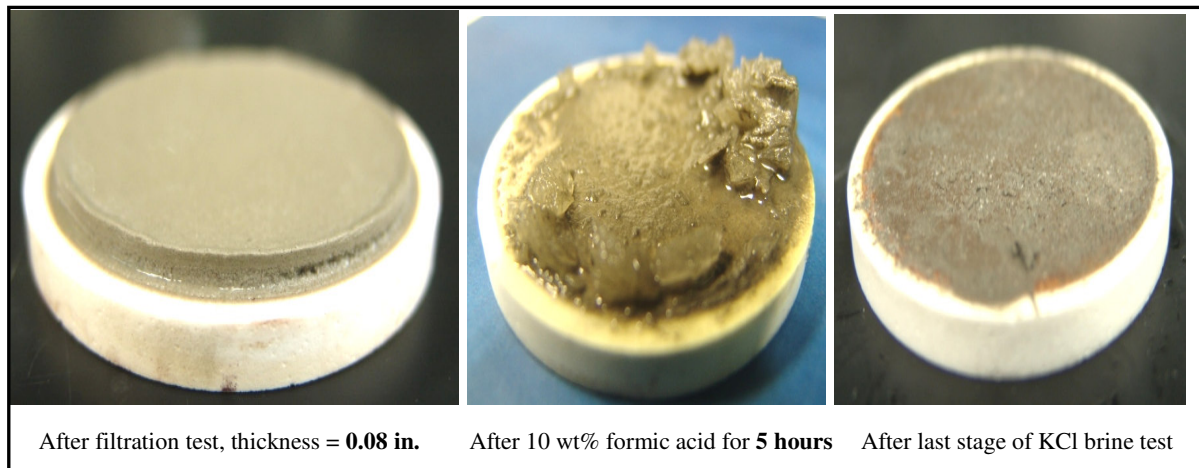
Organic acids are usually added to potassium formate brine to dissolve the filter cake solids as mentioned earlier in the introduction section. A 10 wt% citric acid was prepared in potassium formate brine at temperature of 122°F. At room temperature, the acid formed a sticky ball of solid while mixing the solution and for that reason it was re-prepared at higher temperature. The filter cake thickness was 0.20 in. at 301°F and 200 psi, Figure 55. The citric acid was able to thin the filter cake, but not remove it completely. The removing efficiency after using citric acid was 44 wt%.



**Figure 55:** Filter cake after filtration test and 10 wt% citric acid solution, temperature = 301°F and pressure = 200 psi

#### 4.2.7 10 wt% Formic Acid

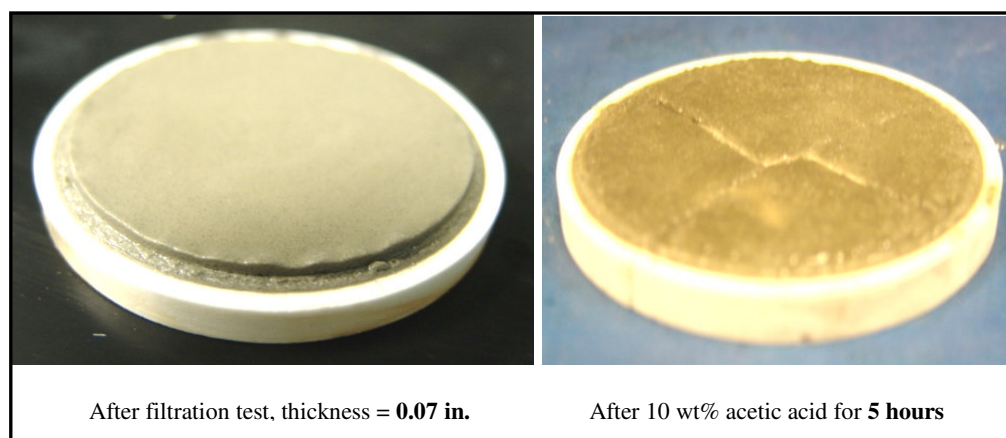
In this experiment, formic acid was tested to remove the filter cake damage. 10 wt% formic acid was added to the potassium formate brine at temperature of 122°F. A corrosion inhibitor was mixed in this and the previous acid solutions to avoid any corrosion to the filter press cell at HPHT. The pH and the density of the fresh solution were 6.6 and 93 pcf, respectively at temperature of 72°F. The formic acid solution was able to remove only 24 wt% of the filter cake solids, Figure 56. The cell was then disconnected after soaking the solution for 5 hrs. The pH of the returned liquid was almost similar to the fresh one, 6.8.



**Figure 56:** Filter cake after filtration test and 10 wt% formic acid solution, temperature = 304°F and pressure = 200 psi

#### 4.2.8 10 wt% Acetic Acid

10 wt% acetic acid was mixed in potassium formate brine at room temperature. The acid showed a good compatibility with the formate brine at room and high temperatures. The solution pH and the density were measured at temperature of 72°F and were equal to 7.35 and 89.27 pcf, respectively. The acetic acid solution was soaked for 5 hrs at temperature and pressure of 300°F and 200 psi. The removing efficiency was measured at the end of the test and was only 28 wt%, Figure 57.

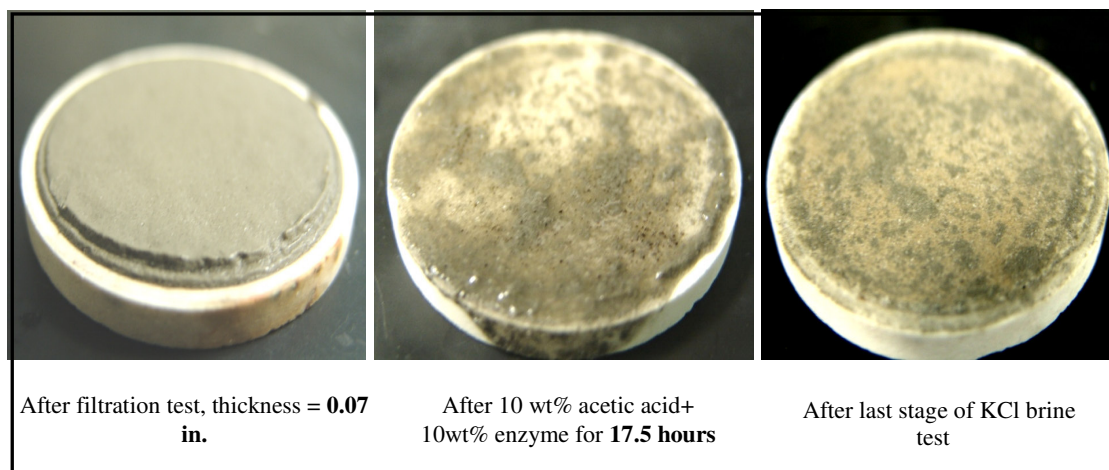


**Figure 57:** Filter cake after filtration test and 10 wt% acetic acid solution, temperature = 300°F and pressure = 200 psi

#### 4.2.9 10 wt% Acetic Acid Mixed with 10 wt% Cellulose Enzyme

Previous experiments showed that organic acids alone can not remove and dissolve most of the filter cake damage. Polymers coated the calcium carbonate particles and that will act as a barrier between the acids and the particles. 10 wt% cellulose enzymes and 10 wt% acetic acid were mixed properly with PFB solution at room temperature. The mixture was compatible at room temperature and no precipitate was noticed. The temperature of the cell was fixed on 242°F because most of the enzymes fluids do not work at temperatures above 250°F and that might cause some precipitations. The mixture was left soaking in the cell for 17.5 hours because the enzymes need enough time to have a complete reaction with the polymers. After most of the cellulose polymer chains were broken, the acetic acid start the reaction with the calcium carbonate particles that covered previously by polymers. The removing efficiency result was excellent and more than 85% of the filter cake was successfully removed, Figure 58. Xanthan enzyme

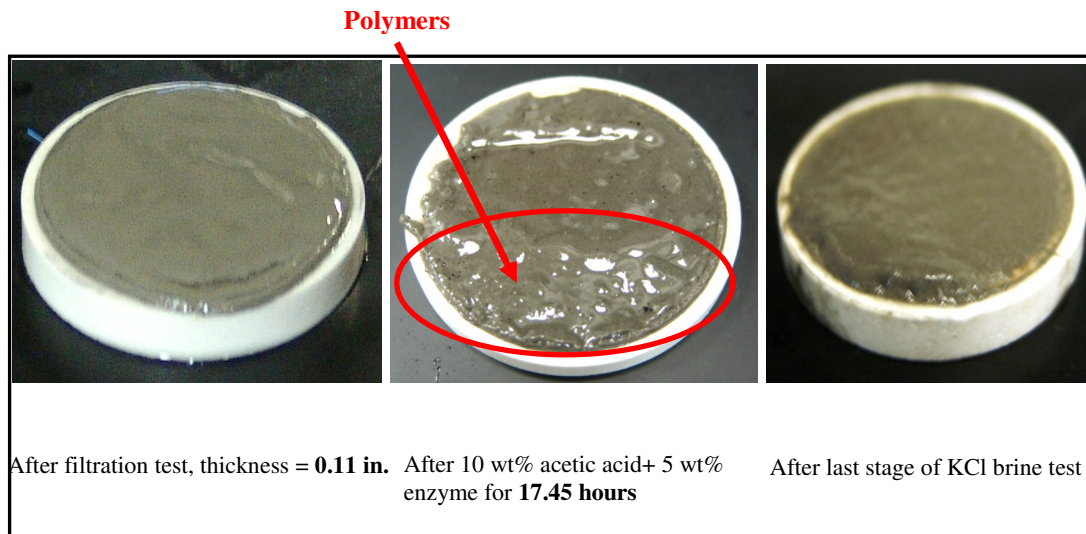
was not used in this experiment because most of the polymers that retained on the filter cake were cellulose since its function was reducing the drilling fluid losses.



**Figure 58:** Filter cake after filtration test and 10 wt% acetic acid mixed with 10 wt% enzyme solution, temperature = 242°F and pressure = 220 psi

#### *4.2.10 10 wt% Acetic Acid Mixed with 5 wt% Cellulose Enzyme*

The objective of this experiment is to determine the effect of 5 wt% cellulose enzyme in acetic acid solution on the polymeric materials and  $\text{CaCO}_3$  compared to the previous test (10 wt% enzyme). Figure 59 shows that the 5 wt% enzyme solution was not able to breakdown most of the polymer chains to lower sugar units. The soaking time was almost equal for both tests. The removing efficiency, based on initial and final brine flow rate, was below 40%. So, 10 wt% cellulose enzyme mixed with 10 wt% acetic acid solution is the optimum cleaning formula to remove the polymer and dissolve the calcium carbonate particles in the filter cake layer.



**Figure 59:** Filter cake after filtration test and 10 wt% acetic acid mixed with 5 wt% enzyme solution, Temperature = 251°F and pressure = 200 psi

### 4.3. Surface Tension Experiments

The PFB solution surface tension is very important to be measured since it is used both in drilling, completion, and workover fluids. The surface tension of the acids solution will be changed when mixed with PFB fluid. Surface tension measurements were done at wide range of temperatures and at a pressure of 72 psi.

#### 4.3.1 PFB solution

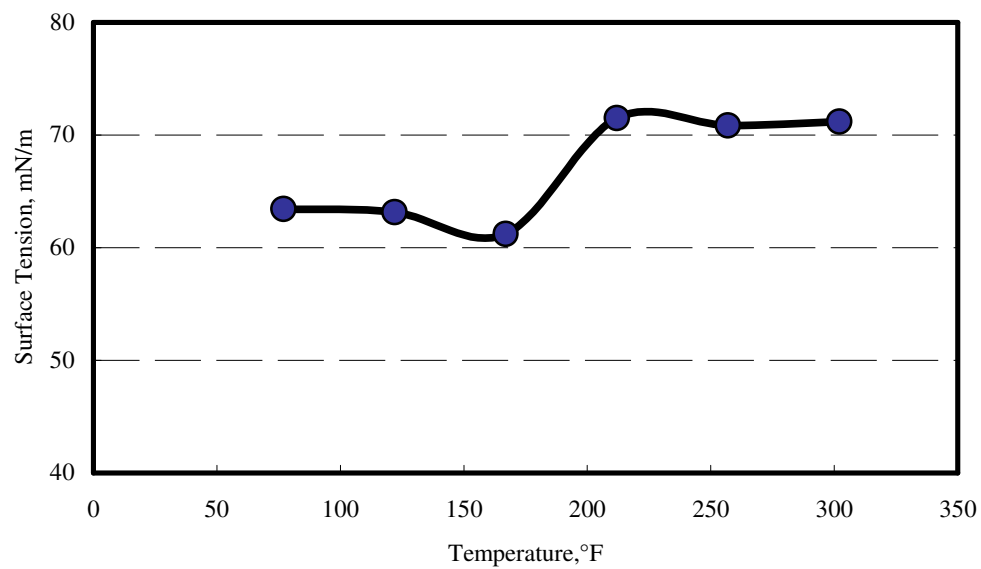
The PFB fluid was used as received from the field without any dilution. The density was measured and equal to 94.8 pcf at room temperature. The surface tension was measured at a wide range of temperature condition. At room temperature, the PFB solution gave



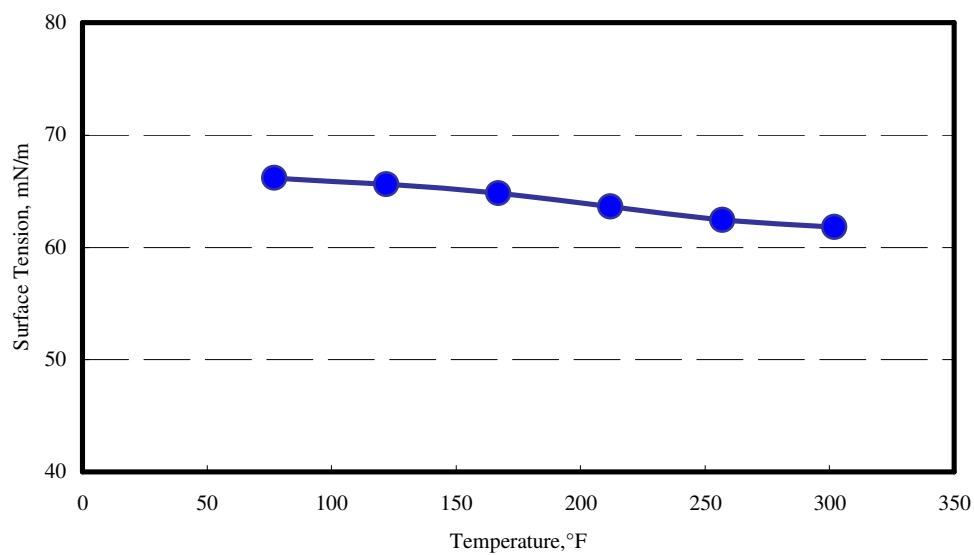
63.7 mN/m of surface tension. As temperature increased to 122°F, the surface tension decreased slightly to 63.1 mN/m. The general trend of the surface tension was increased, as temperature increased above 200°F. Figure 60 shows that behavior and it is up normal because the surface tension usually decreased as temperature increased. The surface tension experiment was repeated several times to make sure there was no experimental error; the same behavior was noticed in all experiments. The instrument was calibrated in each test by measuring the surface tension of the water.

#### *4.3.2 10 wt% Formic Acid Prepared in PFB Solution*

A 10 wt% formic acid was mixed in PFB solution at 130°F to avoid any solid precipitation at room temperature condition. The sample was loaded in the instrument to measure its' surface tension at high temperatures. The surface tension at room temperature was 66.2 mN/m. Then, it decreased, as temperature increased above 122°F as shown in Figure 61.



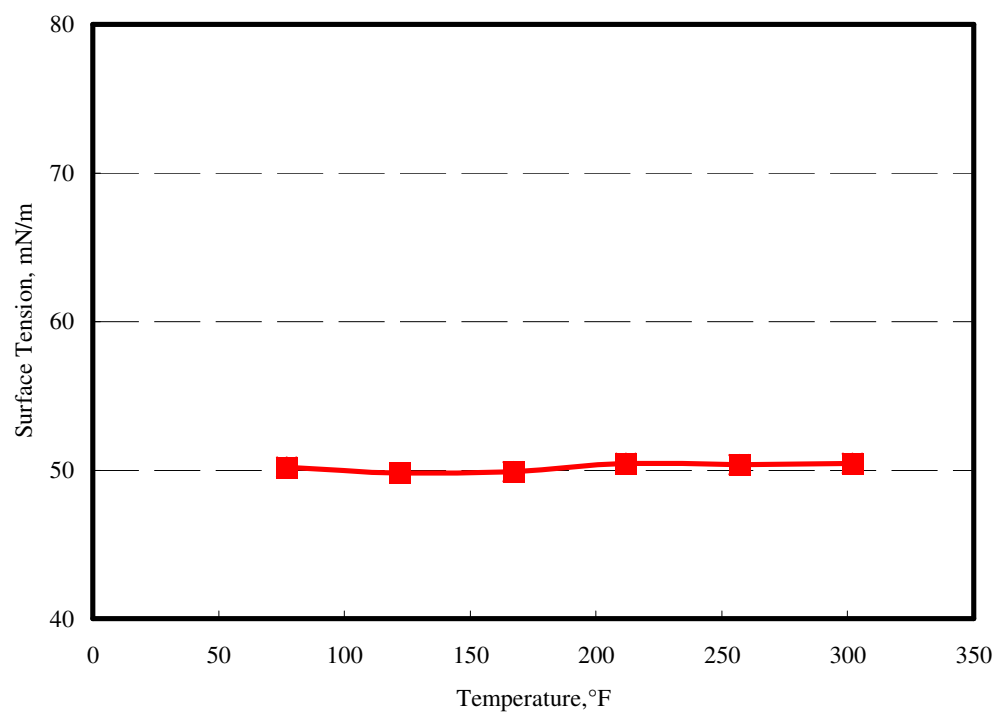
**Figure 60:** Surface tension of PFB at high temperature



**Figure 61:** Surface tension of 10 wt% formic acid at high temperature

#### 4.3.3 10 wt% Acetic Acid Prepared in PFB Solution

Acetic acid, as mentioned in chapter III, was not precipitating when mixed with PFB solution at room temperature. A 10 wt% acetic acid was prepared and then the surface tension was measured. The surface tension was equal to 50 mN/m and then it remained almost constant as temperature increased, Figure 62.



**Figure 62:** Surface tension of 10 wt% acetic acid at high temperature

## CHAPTER V

### CONCLUSIONS

Based on the experimental results and analysis of the data, the following conclusions may be drawn.

1. The DFF was compatible with 10 vol% ethyl lactate ester at room temperature and 300°F.
2. Mixing DFF with UFB at different ratios showed solid precipitates at room conditions.
3. A significant amount of settled solids were observed when PFB was mixed with UFB at ratio 50:50 and at room and high temperatures.
4. Citric and formic acids at 10 wt% were incompatible with PFB; they resulted in some suspended and settled solids at room temperature.
5. No solid precipitate was noticed when citric and formic acids were mixed with PFB at high temperature.
6. A 10 wt% of acetic acid was compatible with PFB at ambient and high temperature conditions.
7. A phase separation was noticed when ethyl lactate ester was mixed with PFB solution.
8. Chelating agent and enzymes solution were compatible with PFB solution.

9. A 10 vol% of ethyl lactate ester was prepared in KCl brine, and it was compatible. Excellent filter cake removing efficiency was attained by the generated lactic acid.
10. Lab experiment indicated that free-solids formate brine can cause some erosion to the filter cake damage and will not remove filter cake completely.
11. Organic acids were not able to cleanup the filter cake damage efficiently. The calcium carbonate particles were covered by the polymers and thus acted as a barrier to the acids to react with the calcite particles.
12. Polymers backbone and side chains in the filter cake can be broken by using specific enzymes.
13. Enzymes-acetic acid solution gave the highest removing efficiency result compared to other cleaning fluids.
14. The surface tension of formic acid was the highest compared to lactic, acetic acids and potassium formate brine.

**NOMENCLATURE**

MRC	Maximum reservoir contact
DIF	Drill-in fluid
WBM	Water-based mud
OBM	Oil-based mud
ECD	Equivalent circulation density
DFE	Drilling fluid filtrate
wt%	Weight percent
vol.%	Volume percent
UFB	Unyazah formation brine
PAC	Polyanionic cellulose
PFB	Potassium formate brine
UFB	Unyazah formation brine
HPHT	High pressure high temperature
pcf	lb/ft <sup>3</sup>
psi	lb/in <sup>2</sup>

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