THICKNESS MEASUREMENT OF FRACTURE FLUID GEL FILTER CAKE
AFTER STATIC BUILD UP AND SHEAR EROSION

A Thesis
by
BEN XU

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 2010

Major Subject: Petroleum Engineering
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Approved by:

Chair of Committee, A. Daniel Hill
Committee Members, Ding Zhu
Yuefeng Sun
Head of Department, Stephen A. Holditch

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ABSTRACT

Thickness Measurement of Fracture Fluid Gel Filter Cake After Static Build Up and Shear Erosion. (May 2010)

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Chair of Advisory Committee: Dr. A. Daniel Hill

The hydraulic fracturing treatment is an essential tight sand gas reservoir stimulation that employs viscous fluid to break the formation rock to create a fracture and transport the propping agent to support the fracture from naturally healing. Despite proven economic benefit, the hydraulic fracture fluid damages the producing formation and the propped fracture. To analyze the gel damage effect quantitatively, the filter cake thickness is used as a parameter that has not been measured before.

This project was divided into two stages. The first stage built up a filter cake and measured the filter cake thickness by a laser profilometer. A correlation between leakoff volume and filter cake thickness was produced. The second stage eroded the filter cake by flowing original fracturing fluid through the core sample to study the fracturing fluid shear clean up effect on filter cake thickness.

The filter cake was built up in the lab and the thickness was measured with different methods. The profilometer has been tested as an effective tool to measure the filter cake thickness. A correlation for crosslinked guar fracture fluid filter cake
thickness was produced. An experiment setup used to shear erode the filter cake was built and tested. The results showed the filter cake was not eroded at 200 s\(^{-1}\) shear rate.
For Julia
ACKNOWLEDGEMENTS

My appreciation goes to my committee chair, Dr. Dan Hill, and my committee members, Dr. Ding Zhu and Dr. Yuefeng Sun, for their guidance and support throughout the course of this research. I also want to thank the Texas A&M University Petroleum Engineering Department for funding my research and study. I also want to extend my gratitude to Mr. Joseph Ayoub from Schlumberger, who supported this research, and to all my friends and colleagues.

Finally, thanks to my mother and father for their encouragement and to my wife for her patience and love.
# TABLE OF CONTENTS

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>ABSTRACT</td>
<td>iii</td>
</tr>
<tr>
<td>DEDICATION</td>
<td>v</td>
</tr>
<tr>
<td>ACKNOWLEDGEMENTS</td>
<td>vi</td>
</tr>
<tr>
<td>LIST OF FIGURES</td>
<td>ix</td>
</tr>
<tr>
<td>LIST OF TABLES</td>
<td>xi</td>
</tr>
<tr>
<td>1. INTRODUCTION</td>
<td>1</td>
</tr>
<tr>
<td>1.1 Gel Damage in Hydraulic Fracturing</td>
<td>1</td>
</tr>
<tr>
<td>1.2 Literature Review</td>
<td>3</td>
</tr>
<tr>
<td>1.3 Research Objective</td>
<td>6</td>
</tr>
<tr>
<td>2. EXPERIMENTAL APPARATUS AND DESIGN</td>
<td>7</td>
</tr>
<tr>
<td>2.1 Filter Cake Build Up Experimental Design</td>
<td>7</td>
</tr>
<tr>
<td>2.2 Filter Cake Build Up and Erosion Modeling</td>
<td>10</td>
</tr>
<tr>
<td>2.3 Filter Cake Erosion Experiment Apparatus</td>
<td>11</td>
</tr>
<tr>
<td>2.4 Filter Cake Thickness Measurement</td>
<td>11</td>
</tr>
<tr>
<td>2.5 Leakoff Modeling and Calculation</td>
<td>13</td>
</tr>
<tr>
<td>3. EXPERIMENTAL PROCEDURE</td>
<td>15</td>
</tr>
<tr>
<td>3.1 Filter Cake Build Up Experimental Procedure</td>
<td>15</td>
</tr>
<tr>
<td>3.1.1 Leaking Problem Elimination</td>
<td>18</td>
</tr>
<tr>
<td>3.1.2 O-ring Installation</td>
<td>20</td>
</tr>
<tr>
<td>3.2 Core Sample Preparation</td>
<td>20</td>
</tr>
<tr>
<td>3.3 Core Sample Saturation</td>
<td>21</td>
</tr>
<tr>
<td>3.4 Fracturing Fluid Mixture Procedure</td>
<td>21</td>
</tr>
<tr>
<td>3.5 Fluid Rheology Measurement</td>
<td>23</td>
</tr>
<tr>
<td>3.6 Rock Permeability and Porosity Measurement</td>
<td>25</td>
</tr>
<tr>
<td>3.7 Shear Erosion Experimental Procedure</td>
<td>26</td>
</tr>
<tr>
<td>3.8 Profilometer Thickness Measurement</td>
<td>28</td>
</tr>
<tr>
<td>3.9 Micrometer Thickness Measurement</td>
<td>28</td>
</tr>
</tbody>
</table>
4. RESULTS AND DISCUSSION ................................................................................. 30
   4.1  Thickness Measurement.................................................................................... 30
       4.1.1 Verification of the Profilometer Thickness.................................................. 30
       4.1.2 Micrometer Thickness Comparison ............................................................... 32
       4.1.3 Scan Resolution.......................................................................................... 34
       4.1.4 Core Deformation....................................................................................... 34
       4.1.5 Removal of Filter Cake ................................................................................ 35
   4.2  Correlation Between Leakoff Volume and Filter Cake Thickness .................... 35
   4.3  Shear Eroded Filter Cake Thickness.................................................................... 37
   4.4  Leakoff Coefficient .......................................................................................... 41
   4.5  Filter Cake Thickness Coefficient........................................................................ 42

5. CONCLUSIONS AND RECOMMENDATIONS....................................................... 45
   5.1  Conclusions ..................................................................................................... 45
   5.2  Future Work Recommendations.......................................................................... 45

NOMENCLATURE............................................................................................................ 47

REFERENCES.................................................................................................................. 49

VITA .................................................................................................................................. 51
# LIST OF FIGURES

<table>
<thead>
<tr>
<th>Figure</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Figure 2.1</td>
<td>Fracturing Fluid Pumping and Mixing Setup</td>
<td>8</td>
</tr>
<tr>
<td>Figure 2.2</td>
<td>Filter Cake Build Up Apparatus</td>
<td>9</td>
</tr>
<tr>
<td>Figure 2.3</td>
<td>Fracturing Fluid Shear Erosion Setup</td>
<td>11</td>
</tr>
<tr>
<td>Figure 2.4</td>
<td>Profilometer Components (Malagon 2006)</td>
<td>12</td>
</tr>
<tr>
<td>Figure 2.5</td>
<td>Profilometer Data Scanning Path (Malagon 2006)</td>
<td>13</td>
</tr>
<tr>
<td>Figure 3.1</td>
<td>Filter Cake Build Up Procedure Diagram</td>
<td>16</td>
</tr>
<tr>
<td>Figure 3.2</td>
<td>Filter Cake Build Up Setup</td>
<td>16</td>
</tr>
<tr>
<td>Figure 3.3</td>
<td>Leakoff Fluid</td>
<td>19</td>
</tr>
<tr>
<td>Figure 3.4</td>
<td>Crosslinked Fracturing Fluid</td>
<td>23</td>
</tr>
<tr>
<td>Figure 3.5</td>
<td>Grace M5600 Rheometer</td>
<td>24</td>
</tr>
<tr>
<td>Figure 3.6</td>
<td>Fracturing Fluid Rheology Diagram</td>
<td>25</td>
</tr>
<tr>
<td>Figure 3.7</td>
<td>Peristaltic Pump</td>
<td>27</td>
</tr>
<tr>
<td>Figure 4.1</td>
<td>FS06B Filter Cake Side View</td>
<td>31</td>
</tr>
<tr>
<td>Figure 4.2</td>
<td>Profilometer Scan of a Lacerated Filter Cake at 0.025 Inch Resolution</td>
<td>32</td>
</tr>
<tr>
<td>Figure 4.3</td>
<td>Thickness Verification by Micrometer</td>
<td>33</td>
</tr>
<tr>
<td>Figure 4.4</td>
<td>Micrometer and Profilometer Thickness Comparison</td>
<td>34</td>
</tr>
<tr>
<td>Figure 4.5</td>
<td>Correlation of Leakoff Volume Versus Filter Cake Thickness</td>
<td>36</td>
</tr>
<tr>
<td>Figure 4.6</td>
<td>Shear Eroded Filter Cake Thickness Plotted with Original Filter Cake Thickness Correlation</td>
<td>38</td>
</tr>
<tr>
<td>Figure 4.7</td>
<td>Filter Cake after 200 s⁻¹ Shear Rate Erosion</td>
<td>39</td>
</tr>
<tr>
<td>Figure 4.8</td>
<td>Stretched Filter Cake after 200 s⁻¹ Shear Rate Erosion</td>
<td>40</td>
</tr>
<tr>
<td>Figure 4.9</td>
<td>Leakoff Volume versus Leakoff Time Correlation for Each Experiment</td>
<td>41</td>
</tr>
<tr>
<td>Figure 4.10</td>
<td>Illustration of Concentration Factor</td>
<td>43</td>
</tr>
<tr>
<td>Figure 4.11</td>
<td>Concentration Factor Correlation</td>
<td>44</td>
</tr>
</tbody>
</table>
### LIST OF TABLES

<table>
<thead>
<tr>
<th>Table</th>
<th>Description</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>Table 1.1</td>
<td>Filter Cake Effect on the Conductivity (McDaniel and Parker 1988)</td>
<td>4</td>
</tr>
<tr>
<td>Table 3.1</td>
<td>Fracturing Fluid Recipe</td>
<td>22</td>
</tr>
<tr>
<td>Table 3.2</td>
<td>Filter Cake Shear Erosion Experiment Parameters</td>
<td>27</td>
</tr>
<tr>
<td>Table 4.1</td>
<td>Thickness Measuring Results</td>
<td>37</td>
</tr>
<tr>
<td>Table 4.2</td>
<td>Filter Cake Shear Erosion Results</td>
<td>38</td>
</tr>
<tr>
<td>Table 4.3</td>
<td>Leakoff Coefficient</td>
<td>42</td>
</tr>
</tbody>
</table>
1. Introduction

1.1 Gel Damage in Hydraulic Fracturing

Hydraulic fracturing is an oil and gas production stimulation method, which uses fluid to break the formation rock, to create a fracture and transport solid particles to support the fracture from natural healing. Despite proven economical benefit, the hydraulic fracture technique damages the producing formation as well as the propped fracture. Among all the low permeability reservoir stimulation techniques including horizontal drilling, acidizing etc, by changing the reservoir flow pattern, the reservoir-penetrating fractures alter the flow path of fluid from the reservoir to the well bore and increase the economical production rate. The flow path will penetrate the drilling and completion-induced damage zone. But more importantly, the fluids will flow through the low permeability matrix shortly before flowing through the high conductivity fracture and entering the wellbore. The wells completed in unconventional gas reservoirs, such as tight sand gas, coal-bed methane and shale gas, would not have an economically adequate production rate until they have been fractured hydraulically.

Hydraulic fracture operations contain the process of pad, slurry and flush. In the first stage, called the pad, a clean fracturing fluid is injected into the reservoir at a pressure higher than the formation fracture gradient. The pads create an unpropped fracture.

This thesis follows the style of SPE Journal.
This initiated fracture provides adequate width around the wellbore region to allow proppant-laden slurry to flow into the fracture without screen out. The fluid volume that is used to initiate a fracture is called pad volume.

After the fracture has been formed, the proppant-laden slurry is pumped into the fracture and holds the fracture against the fracture closure stress. The slurry carries the proppant to the tip of the fracture. Ideally, the proppant shall fill all the fracture space just as all the pad fluid is leaked off into the reservoir. The slurry would extend the fracture further. When the proppant was carried deep into the fracture, the fracturing fluid was leaking off into the formation in the meantime. The first portion of slurry, which has the lowest proppant concentration, would be concentrated. Meanwhile the last portion of slurry has the highest proppant concentration. In this way, the proppant would cover the fracture face as evenly as possible.

After all the proppant has been pumped into the fracture, the well is shut down and fracture fluid breaks down to low viscosity fluid. The proppants are left in the fracture and hold up the fracture open.

A large amount of proppant needs to be transported by the highly viscous fluid into the fracture. Consequently, making a higher viscosity linear gel increases the polymer concentration. Unbroken polymers left in the fracture. These residual unbroken polymers are the cause of permeability impairment in the proppant pack and conductivity damage in the hydraulic fracture. This gel damage mechanism is also the cause of decreasing effective fracture length, which yields even lower effective fracture conductivity. Lower conductivity yields lower well productivity.
In the 1970s, the crosslinked gel water based fracturing fluid became widely used and was considered a breakthrough in fluid technology. Crosslinked fluid required lower polymer concentrations to obtain the designed viscosity. Other than base fluid, specific fluid functions and attributions were achieved by adding many additives such as fluid-loss additive, stabilizer and breaker. Different reservoir conditions such as high temperature, clay swelling and long pumping time also needed specialized additives.

1.2 Literature Review

B.B. Williams (1970) presented a theory that divided the leakoff into three steps. In the first period, the leakoff fluid displaced the reservoir fluid. The leakoff rate is high initially but decreased to a steady speed when steady is reached. Filter cake did not build up, and the leakoff is controlled by the formation permeability alone. In the second time step, filter cake begun to build up and the leakoff speed decreased. In the final step, the filter cake thickness stopped to develop until the shear stress equaled the filter cake yield stress. The fluid flowing between the fractures etched the filter cake.

McDaniel and Parker (1988) presented experiment results that demonstrated filter cake could decrease conductivity to 20% of original at 6000 psi closure stress. This paper did not include the filter cake thickness. Table 1.1 is the results.
Table 1.1  Filter Cake Effect on the Conductivity (McDaniel and Parker 1988)

<table>
<thead>
<tr>
<th>Closure Stress</th>
<th>Long-term conductivity with filter cake</th>
<th>Long-term conductivity without filter cake</th>
<th>Conductivity remain</th>
</tr>
</thead>
<tbody>
<tr>
<td>psi</td>
<td>md-ft</td>
<td>md-ft</td>
<td>%</td>
</tr>
<tr>
<td>2000</td>
<td>1400</td>
<td>3500</td>
<td>40</td>
</tr>
<tr>
<td>4000</td>
<td>520</td>
<td>2200</td>
<td>23</td>
</tr>
<tr>
<td>6000</td>
<td>230</td>
<td>950</td>
<td>24</td>
</tr>
</tbody>
</table>

Prud’Homme and Wang (1993) did an interesting experiment to measure the filter cake thickness versus the gel concentration. However, they used a membrane instead of core sample to build up the filter cake, so the results do not apply directly to the field. They proposed that the filter cake growth in thickness is controlled by the shear stress exerted by the fracturing fluid flowing through the wall and the yield stress of the filter cake. The cake growth would stop if the fluid shear stress becomes equal to the filter cake yield stress. The erosion begins when the fluid stress is larger than the filter cake yield stress.

\[
\frac{dM}{dt} = \alpha q, \tau_w < \tau_y \\
= 0, \tau_w = \tau_y \\
= \text{rate of erosion, } \tau_w > \tau_y
\]  \hspace{1cm} (1.1)

Navarrete, R. C. et al. (1994) produced a correlation between shear rate and fluid loss coefficient. His results showed that the effect of shear rate is to increase the spurt loss and leakoff rate. However, the magnitude of shear rate did volume affect the spurt loss volume and leakoff rate very much. He tried to measure the filter cake thickness during leakoff process.
Vitthal, S. and McGowen, J.M. (1996) conducted more than 1000 laboratory experiments to study the effects of shear rate, permeability differential pressure, temperature, gel concentration, fluid-loss additives and fluid type. They found that dynamic fluid loss is significantly higher than static fluid loss. The linear gels fluid loss is sensitive to permeability and pressure, and a crosslinked gel is sensitive to shear rate. The fluid loss could be modeled through the mechanisms of non-Newtonian viscous invasion model, which was developed by Settari (1985). Even though they nearly studied every aspect of fracturing fluid leakoff properties, filter cake thickness and shear erosion effect was not included.

Ayoub et al. (2006a) presented an extensive filter cake study on borate crosslinked gel. They first confirmed the yield stress behavior of the fracturing fluid with fracture initiation gradients as high as 14 psi/ft corresponding to a yield stress of 17 Pa. They found that the oxidizer breaker could not reduce yield stress effect for the cases where the cumulative filter cake thickness equal or approaches the propped width. They concluded that the polymer concentrated in the filter cake solely, which needs to confirm through this study.

Wang Y. and Holditch S.A. (2009) simulated the fracture clean up process and considered the filter cake and assumed a high yield stress. Their simulation results suggest that the filter cake would not reduce the cumulative production if the filter cake thickness accounts for less than 25% of the fracture width. However, the thickness number they use tends to be very small compare to this study.
1.3 Research Objective

The research objective is listed below:

1. Design filter cake experimental apparatus and procedure.
2. Model filter cake build up and erosion process.
4. Measure filter cake thickness by profilometer.
2. EXPERIMENTAL APPARATUS AND DESIGN

The filter cake thickness build up and measurement process is composed of three procedures: pumping fracture fluid into the conductivity cell, closing and pressurizing the fracturing fluid to leak off, and using a profilometer to scan the filter cake.

The shear erosion experiment is based upon the filter cake thickness experiment.

2.1 Filter Cake Build Up Experimental Design

This project is divided into two stages. The first stage is designed to build up filter cake and measure the filter cake thickness with a laser profilometer. A correlation of leakoff volume versus filter cake thickness is produced. The second stage is meant to erode a filter cake by fracturing fluid to study the filter cake clean up effect.

Three equipment groups comprise the filter cake build up apparatus:

- Gel mixing and pumping
- Static fluid leakoff cell
- Profilometer and filter cake thickness micrometer

The gel mixing and pumping apparatus consists of the following equipments:

- Mixing tank
- Multistage centrifugal pump
- Modified API conductivity core holder
- Ultrasonic Flow meter
- Closure stress transmitter
- Closure stress frame
- Leakoff volume collector
- Waste fluid vessel

The schematic of the equipments is shown in Figure 2.1.

Figure 2.1 Fracturing Fluid Pumping and Mixing Setup

The base fluid was prepared in the mixing tank before being fed into the multistage centrifugal pump, and entering the conductivity cell. Crosslinker and other chemicals were
added into the line by a metering pump at a certain speed on the fly. A valve installed at downstream of a high-pressure waste vessel controlled the pumping pressure. While the fluid was flowing through the pressure cell, the flow rate could be measured by an ultrasonic flow meter. A pressure transmitter recorded the flowing pressure, which also is the leakoff pressure.

After the gel is pumped, the cell would be closed and pressurized. The frame applied fixed closure stress during the shut-in period from 0.5 hr to 24 hr. Figure 2.2 is the schematic of the closure stress applying apparatus.

![Figure 2.2 Filter Cake Build Up Apparatus](image-url)
2.2 Filter Cake Build Up and Erosion Modeling

The filter cake thickness is controlled by the shear stress exerted by the tangential flowing fracturing fluid along the wall and the yield stress of the filter cake. The cake growth would stop if the fluid shear stress becomes equal to the filter cake yield stress. The erosion begins when the fluid stress is larger than the filter cake yield stress.

\[
\frac{dM}{dt} = \omega q, \tau_w < \tau_y \\
= 0, \tau_w = \tau_y \\
= \text{rate of erosion}, \tau_w > \tau_y
\]  

(2.1)

where \( M \) is the mass/area of the filter cake, \( \omega \) is the mass fraction of solids in the fluid, \( q \) is the transverse flow rate, \( \tau_w \) is the yield stress of the filter cake wall and \( \tau_y \) is the shear rate exerted on the filter cake by fracturing fluid.

\( \tau_y \) is calculated from the rheology of the fluid. Herschel-Bulkley model represented the relationship between the shear rate and shear stress precisely.

\[
\tau_w = \tau_{yield} + Ky^n_w
\]  

(2.2)

By Navarrete, R. C. (1994), the relationship between flow rate and shear rate is given by,

\[
Q = \frac{\gamma_w w^2 h}{(4 + \frac{2}{n})}
\]  

(2.3)
where $K$ is the consistency factor, $n$ is the behavior index, $w$ is the width of the fracture, $h$ is the fracture height, $\tau_w$ is the shear rate at the filter cake wall.

2.3 **Filter Cake Erosion Experiment Apparatus**

The shear rate on the filter cake ranged from 20 to $200 \text{ s}^{-1}$. Before each experiment, the fracturing fluid was taken to a rheometer to get $n'$ and $k'$.

Figure 2.3 is the setup apparatus.

![Fracturing Fluid Shear Erosion Setup](image)

**Figure 2.3** Fracturing Fluid Shear Erosion Setup

2.4 **Filter Cake Thickness Measurement**

The profilometer measured the thickness of the filter cake before and after erosion. This profilometer has been tested and validated as a useful instrument to measure the filter cake thickness.

Four parts compose this profilometer: Laser Displacement Sensor, Controller Box, Distance Transducers and Stepping Motor. Figure 2.4 is illustration of the profilometer components.
A profilometer measures small distance variations from the object to the sensor. A surface area is moved along its length on a moving table and the sensor is positioned above the sample to measure the vertical distance with a certain resolution. The laser displacement sensor is from Acuity Laser Measurement. The sensor uses triangulation to measure distance. It emits a laser beam and captures the reflection by an array of sensors. The area of the reflection is then processed to the distance data. This sensor has an accuracy of 0.002 in (0.005 cm), and its measurement range is 1 inch (2.54 cm). The horizontal resolution is 0.1 inch, and the scanning time is 11 minutes.

The stepping motor moves the table in the X and Y direction. These motor units are high-torque, low-vibration devices attached to each of the milling table directions. The controller box moves the X direction forward and backward during the data reading, and another motor moves the table in small steps in the Y direction after each direction change in X. There is an illustration of the scanning pattern in Figure 2.5.
Two magnetic elements attached to the sample table for X and Y direction displacement measurement. The magnetic wave sensors measure the horizontal displacement.

All the moving and measuring parts are controlled by the controller box and Labview software. A Data Acquisition Board controls the entire system. This board received data from the laser sensors and the micro-pulse transducer sensors. The data is transferred to the Labview software.

2.5 Leakoff Modeling and Calculation

Carter (1957) presented a filter cake leakoff model.

The general relationship between leakoff time and leakoff volume is defined as:

$$u_L = \frac{C_w}{\sqrt{t}}$$  \hspace{1cm} (2.4)

where the $u_L$ is defined as:

$$u_L = \frac{dV}{dt}$$  \hspace{1cm} (2.5)
Integration (2.5) about time $t$:

$$ \int u_L \, dt = \int \frac{C_w}{\sqrt{t}} \, dt = 2C_w \sqrt{t} \quad (2.6) $$

which is the total leakoff volume at a certain point.

Multiply by the total leakoff area $A$, yields the total leakoff volume:

$$ V_L = 2AC_w \sqrt{t} \quad (2.7) $$
3. EXPERIMENTAL PROCEDURE

The filter cake build up experiments and the filter cake shear erosion experiments are the two main parts of this study. The filter cakes build up experiment spanned from 0.5 hour to 24 hours to build up filter cake and produced a correlation between filter cake thickness and leakoff volume. The filter cake shear erosion experiment studied the shear erosion effect on the filter cake thickness.

3.1 Filter Cake Build Up Experimental Procedure

The cores were water saturated before the filter cake was built by leaking off the 40lb/1000 gallon cross-linked Hybro guar gel provided by Halliburton. Prior to the gel was pumped into the core-loaded cell, the O-ring, which used to seal the gap between core and cell, passed the tightness test at 1000-psi pressure. Then, the gel was pumped through the cell at 50-psi flowing pressure without leakoff fluid collected. After 10 minutes, the gel flow was cut off and the cell was closed. Without touching each other, the upper core was pushed into the cell at 500-psi closure pressure to a certain depth. Leakoff fluid was collected during this process. Figure 3.1 is the experimental procedure.
After the filter cake was built, the gel concentration in the filter was assumed higher than 40lb/1000 gallon and was hard to move compared to uncondensed gel.

Figure 3.1 Filter Cake Build Up Procedure Diagram

Figure 3.2 Filter Cake Build Up Setup
Water was flowed through the cell to displace residual fracturing fluid and push out the core without damaging the filter cake. Figure 3.2 is the experimental apparatus.

The profilometer scan takes 11 minutes to finish. For thickness measurement purpose, the measuring points are set 0.1 inch apart from each other. Depending on the resolution one needs, the scanning time could take up to 4 hours.

Following is the filter cake build up experiment procedure:

1. Prepare the core according to Section 3.2.
2. Saturate molded core.
3. Put the core into the cell and use O-ring to seal the gap between the core and conductivity cell.
4. Put the conductivity cell into the hydraulic load frame and use the piston cover to adjust the fracture width to 3/8 inch.
5. Make sure the conductivity cell is at horizontal position.
6. Apply closure stress on the conductivity cell until the top piston touches the top plate. Close the air regulator on the AP-1, 000 hydraulic oil pump.
7. Connect the pressure transducer to the conductivity cell and use the water sprayer to make sure all connections are tight.
8. Connect the tap water line to the conductivity cell.
9. Mix the fracturing gel according to Section 3.4.
10. Use the centrifugal pump or the peristaltic pump to flow the fracturing fluid into the conductivity cell. Close the side valve of the conductivity cell.
11. Leave the residual fracturing fluid in a drum until filter cake experiment.
12. Measure the fracturing fluid rheology according to Section 3.5.

13. Apply 500-psi closure stress on the conductivity cell by the load frame and open the leakoff line to monitor the leakoff volume.

14. Use the pressure transducer to maintain the leakoff pressure around 500 psi.

15. Wait until 0.5 to 24 hours and take out the core by tap water pressure.

16. Measure the Filter cake thickness by the Micrometer and the Profilometer according to Section 3.8.

### 3.1.1 Leaking Problem Elimination

If the leakoff fluid is not clear, the o-ring between the core and the cell is damaged or un-properly installed. The whole equipment needs to be dissembled and re-install.

Figure 3.3 is an example of clear leakoff fluid. The leakoff fluid is as clear as water, because the core sample filtered out the polymer in the fracturing fluid. These polymers form the filter cake.
Figure 3.3 Leakoff Fluid

The un-clear leakoff fluid is a sign that the data is not reliable. On the other hand, frequently checking the clarity of leakoff fluid makes sure that the experiment is conducted properly. Figure 3.3 is a picture of clear leakoff fluid.
3.1.2 O-ring Installation

The o-ring installation is tricky and easy to go wrong.

The corner of the core sample needs to be filed into a rounded shape to make o-ring sliding into the groove easier.

A special L shape tool made by strong tubing is adapted to reach inside the cell and push the o-ring when necessary. This procedure needs to be illuminated by another person with a hand flash light.

3.2 Core Sample Preparation

Kentucky sandstone was use in the filter cake build up and erosion experiments. The permeability of this rock is 0.2-0.6 md. The rocks were cut into a 7 in. long, 1.65in wide, and 3 in. in height rectangular shape with rounded edges. Silicone rubber covering the rock provided the preliminary sealing agent between the rock and the conductivity cell.

Teflon tape and grease was initially used to prevent the leaks. However, sealing quality is varying from person to person and leakoff pressure stability suffered. Therefore, the API cell was modified to accommodate an O-ring between the cell and core. This modification increased the sealing pressure to more than 1000 psi.

The flowing procedure explains how to prepare the core samples:

1. Rub the modified mold with acetone. Make sure the mold surface is clean.
2. Spray Sprayon S00315 on the mold three times within 3 minutes between each spray.
3. Put the mold pieces together and tighten the screws.
4. Clean the rock sample before molding.
5. Covering the bottom and top surface with blue tape, cut unnecessary tape.

6. Brush the uncovered area with silicone primer SS41501P three times with 15 minutes between brushing.

7. Put the rock into the mold and leave 0.07 in. clearance between mold and core.

8. Pour a mixture of 75 cc silicone potting and 75 cc silicone curing mixture into the syringe.

9. Inject the mixture into the clearance between the core and the mold slowly until the fluid is level to the top.

10. Remove the top tape and put the mold into the oven at 60°C for 2 hours.

11. Take out the rock then proceed to the next experimental step.

3.3 Core Sample Saturation

Before the core was taken into the filter cake build up experiment, water must be filled into the core pore space. The air is removed by a special vacuum pump, and then the water filled into the empty space. This process takes about 4 hours to finish.

3.4 Fracturing Fluid Mixture Procedure

HPG borate fluid was used in this filter cake build up experiment. This a mainstream hydraulic fracture fluid popularly used in US market. All the experiments are conducted at room temperature.
Table 3.1 is the recipe of this fluid.

### Table 3.1  Fracturing Fluid Recipe

<table>
<thead>
<tr>
<th>Chemical</th>
<th>Concentration</th>
</tr>
</thead>
<tbody>
<tr>
<td>Guar, lb/mgal</td>
<td>30-50</td>
</tr>
<tr>
<td>pH Buffer #1 to pH</td>
<td>6.5</td>
</tr>
<tr>
<td>pH Buffer #2 to pH</td>
<td>10.0</td>
</tr>
<tr>
<td>Breaker, gal/mgal</td>
<td>10</td>
</tr>
<tr>
<td>Breaker activator, gal/mgal</td>
<td>1.0</td>
</tr>
<tr>
<td>Borate crosslinker, gal/mgal</td>
<td>0.9</td>
</tr>
<tr>
<td>Crosslink accelerator, gal/mgal</td>
<td>0.2</td>
</tr>
</tbody>
</table>

The following procedure explains how to prepare it:

1. Use the recipe to calculate and measure the proper amounts of chemical.
2. Clean the mixing tank and fill with the proper amount of water.
3. Add the polymer into the tank and turn on the mixer.
4. Add acidity buffer into the fluid to pH 6.5 and let it age for at least 30 minutes.
5. Add alkalinity buffer into the aged fluid to required pH.
6. Wait until the centrifugal pump and people are coordinated to pump the fluid, **DO NOT DRY RUN THE CENTRIFUGAL PUMP.**
7. Mix crosslinker and accelerator into the reservoir of the metering pump.
8. Add the crosslinker and crosslink accelerator on the fly.
9. Clean the residue fluid in the tank and let the centrifugal pump run for more than 1 hour to clean the fracturing fluid.

A major service company provided the recipe. This fluid could degrade into water after several days. The crosslinked fluid is very viscous and stable. A prepared fluid is illustrated in Figure 3.4.

![Figure 3.4 Crosslinked Fracturing Fluid](image)

3.5 Fluid Rheology Measurement

A Grace M5600 Rheometer measured the fracturing fluid rheology. This rheometer is a couette rotational viscosmeter with high pressure and high temperature capability. Figure 3.5 is an illustration of the rheometer. The rheology data is crucial to the shear rate
calculation on the filter cake face and the yield stress of the filter cake. $K^\prime$, as the flow consistency index and $n^\prime$, as the flow behavior index could be measured from this test.

Following is the procedure to measure the rheology:

1. Prepare 2 gallon fracturing fluid according to Section 3.4.
2. Fill the container with 50 ml fracturing fluid. Overfilling the fluid would produce wrong data.
3. Open the rheology measurement software.

4. Program the rheometer to measure the shear rate from 20 to 200.

5. Output the data into the Excel spread sheet and read the n` and k` number.

Figure 3.6 is the example of the rheology data.

![Figure 3.6 Fracturing Fluid Rheology Diagram](image)

3.6 **Rock Permeability and Porosity Measurement**

Before each experiment, the rock porosity and permeability were measured. The porosity measurements followed this procedure:

1. Measure the core mass $M_D$.

2. Saturate the core with water.

3. Measure the core mass $M_W$. 
4. Use equation \( \phi = \frac{M_{core}}{V_{core}} \frac{\rho_r}{\rho_w - \rho_r} \) to calculate the porosity.

The permeability measurement procedure is listed below:

1. Put the saturated core into the cell.

2. Connect the water faucet to the cell and close the outlet valve.

3. Connect the pressure transducer to the cell.

4. Open the water faucet and check for leakage.

5. If the cell is water tight, open the leakoff valve on the testing core side.

6. Wait until the first drop of water comes out of the leakoff line.

7. Wait for 1 hour of leakoff time and then close the leakoff valve.

8. Measure the leakoff volume.

9. Measure the \( \Delta p \) across the core.

10. Calculate the permeability by Darcy equation.

3.7 Shear Erosion Experimental Procedure

Following picture is the peristaltic pump made by Randolph Austin. This pump is capable to pump high viscosity fluid for the shear erosion experiments. Figure 3.7 is the configuration of the peristaltic pump.
1. Finish the filter cake build up experiment.

2. Prepare fracturing fluid.

3. Setup the experiment cell according to designed parameters in Table 3.2.

<table>
<thead>
<tr>
<th>Name</th>
<th>Leakoff Volume</th>
<th>Shear Erosion Rate</th>
<th>Fracture Width</th>
<th>Flow Behavior index</th>
<th>Flow Rate</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(ml)</td>
<td>(s(^{-1}))</td>
<td>(inch)</td>
<td></td>
<td>(ml/s)</td>
</tr>
<tr>
<td>ES01</td>
<td>90</td>
<td>20</td>
<td>3/8</td>
<td>0.625</td>
<td>10.3</td>
</tr>
<tr>
<td>ES02</td>
<td>89</td>
<td>200</td>
<td>3/8</td>
<td>0.625</td>
<td>104</td>
</tr>
</tbody>
</table>

4. Open the peristaltic pump and flow the fracturing fluid through the cell for 1 hour.
5. Take out the core and measure the thickness.

### 3.8 Profilometer Thickness Measurement

1. Retrieve the core after the filter cake build up process or the filter cake erosion process.
2. Put the core on the center of the scanning table and fasten the table screws to secure the core position.
3. Adjust the laser beam height by controlling the vertical milling table.
4. Make sure the laser range covers the height of the filter cake.
5. Open the computer and open the Labview program named “profilometer.vi”.
6. Reset the position of the table position X and Y to zero, make sure the Z reading from the computer is not smaller than 0.001 inch or larger than 1 inch.
7. Set the $\Delta x$ and $\Delta y$ to 0.1 inch.
8. Wait about 10 minutes for the scan to complete and then remove the filter cake.
9. Repeat the procedure from 1-7, and then proceed to Matlab interface to produce the mean value of the thickness data.

### 3.9 Micrometer Thickness Measurement

1. Clean the RES plate before putting the core sample on it.
2. After the filter cake has been scanned with profilometer, take the core sample to the RES plate.
3. Contact the micrometer probe on the filter cake surface.
4. Move the probe slowly and gently towards filter cake surface without touching it.

5. Reset the micrometer to zero, and then extend the probe through the filter cake to reach the core firmly.

6. Record the data and repeat step 3-5 at 5 more different points.

7. Calculate the arithmetic mean value of these thickness data.
4. RESULTS AND DISCUSSION

4.1 Thickness Measurement

The filter cake thickness measurement is divided into two steps. The first step scans the surface of the filter cake. The second step scans the surface of the core. The difference between both surface scans is the filter cake thickness.

The profilometer is tested as a useful tool to measure the filter cake thickness.

4.1.1 Verification of the Profilometer Thickness

The reliability of thickness data produced by the profilometer was verified by micrometer and other method.

From the side view of the filter cake of the FS06B test as shown in Figure 4.1, the thickness is close to 2 mm. Even though this is an approximation, it provides us a reference number about the thickness.

The filter cake was measured by a micrometer at 6 points to generate a mean thickness value. This number was compared with the profilometer thickness mean value. The micrometer measurement procedure is listed in the section 3.9. This method requires accurate eye alignment of the measure tip on the filter cake surface, thus the accuracy is depended on the individual doing the measurement. The profilometer method is impersonal and fast.
Figure 4.2 illustrates the sensitivity of the profilometer. The upper photograph pictures a lacerated filter cake, which came from an experiment, which failed to protect the filter cake while taking it out from the cell.

This filter cake was scanned at 0.025 inch resolution as shown in the lower picture. The process was according to section 3.8. The profilometer clearly showed the shape of the laceration and the thickened area around the laceration.
Figure 4.2 Profilometer Scan of a Lacerated Filter Cake at 0.025 Inch Resolution

This scan clearly pictured the shape of the filter cake. To verify the thickness data, the arithmetic mean value of the profilometer scan was compared with the micrometer mean value.

4.1.2 Micrometer Thickness Comparison

After the filter cake was scanned by the profilometer, the filter cake was measured with the micrometer at 6 points. The procedure is written in Section 3.9.

Figure 4.3 shows the correct measurement tip position directly above the surface to be measured.
Figure 4.3 Thickness Verification by Micrometer

From the Figure 4.4, the profilometer results show close agreement with the micrometer results, which verified the thickness data.
4.1.3 Scan Resolution

When the $\Delta x$ and $\Delta y$ setted to 0.025 inch, the scanning time was 40 mins. This resolution is not necessary for a thickness measurement and increases the data processing time. The 0.1-0.5 inch $\Delta x$ and $\Delta y$ values are recommended for a accurate thickness measurement.

The scanning time is determined on the scanning resolution of the filter cake. Shorter scanning time also prevented the water evaporation affecting the filter cake.

4.1.4 Core Deformation

The core sample surface was doubted to be deformed under the closure pressure or to be scoured by the fluid. This doubt was eliminated by the profilometer scan comparison.
before and after the experiment. The comparison between both scans showed it has a negligible difference. This means the deformation of the core due to pressure and scouring is below the profilometer vertical resolution.

4.1.5 Removal of Filter Cake

To prevent the breakage of the filter cake before the scan, the filter cake and core has been slowly pushed out from the cell. The redundant fracturing fluid has been removed, and only the filter cake sent to thickness measurement.

During the scan, the core remains on the measurement plate all the time, while the filter cake is removed from the core sample. Otherwise the profilometer scan before and after the filter cake remove would be offset, and errant results would be produced. If this occurred, the whole experiment would start again.

4.2 Correlation Between Leakoff Volume and Filter Cake Thickness

Figure 4.5 shows a linear relationship between leakoff volume and the filter cake thickness. Section 4.5 illustrated the procedures of how to derive a filter cake prediction equation.
Table 4.1 is the results of the filter cake thickness data listed with total leakoff fluid volume. The concentration factor is calculated as following equation.

\[
F_{FC} = \frac{V_f + V_L}{V_f} 
\]

(4.1)

where as, the \( V \) is the total leakoff volume, \( h \) is the filter cake thickness and \( A \) is the total leakoff area. This factor indicates the extent of polymers concentration in the filter cake. As the leakoff volume goes higher, more polymers were concentrated into the filter cake.
cake. Ayoub et al. (2006b) discovered similar effect, and he was observing the average concentration factor in the fracture.

Table 4.1  Thickness Measuring Results

<table>
<thead>
<tr>
<th>Name</th>
<th>Leakoff Volume (ml)</th>
<th>Filter cake Thickness by Profilometer (mm)</th>
<th>Concentration Factor in Filter cake</th>
</tr>
</thead>
<tbody>
<tr>
<td>FS05</td>
<td>31</td>
<td>0.4064</td>
<td>11.6175</td>
</tr>
<tr>
<td>FS08</td>
<td>47</td>
<td>0.5842</td>
<td>10.0018</td>
</tr>
<tr>
<td>FS07</td>
<td>65</td>
<td>0.8128</td>
<td>9.78903</td>
</tr>
<tr>
<td>FS09</td>
<td>68</td>
<td>0.8636</td>
<td>10.7967</td>
</tr>
<tr>
<td>FS10</td>
<td>98</td>
<td>1.0922</td>
<td>11.1549</td>
</tr>
<tr>
<td>FS11</td>
<td>119</td>
<td>1.1938</td>
<td>12.3925</td>
</tr>
<tr>
<td>FS06T</td>
<td>145</td>
<td>1.3462</td>
<td>13.3907</td>
</tr>
<tr>
<td>FS12</td>
<td>179</td>
<td>1.3716</td>
<td>16.2244</td>
</tr>
<tr>
<td>FS13</td>
<td>201</td>
<td>1.4478</td>
<td>17.2596</td>
</tr>
<tr>
<td>FS14</td>
<td>227</td>
<td>1.5494</td>
<td>18.2140</td>
</tr>
<tr>
<td>FS15</td>
<td>243</td>
<td>1.651</td>
<td>18.2980</td>
</tr>
<tr>
<td>FS06B</td>
<td>182</td>
<td>1.4376</td>
<td>14.6033</td>
</tr>
</tbody>
</table>

4.3  Shear Eroded Filter Cake Thickness

By R.C. Navarrete (1994), The following equation relates the shear rate on the surface of the filter cake and the fluid flow rate.

\[
\gamma_w = \frac{q(4 + \frac{2}{n})}{w^2 h} \quad (4.2)
\]

The Grace M5600 rheology meter measures the fracturing fluid rheology data. Table 4.2 shows the results for the shear erosion experiments. Figure 4.6 illustrates the leakoff volume correlation with the shear eroded filter cake thickness and the filter cake
thickness. The red dots in the Figure 4.6 are the filter cake thickness data for shear rates at 20 and 200 s\(^{-1}\).

<table>
<thead>
<tr>
<th>Name</th>
<th>Leakoff Volume (ml)</th>
<th>Shear Erosion Rate (s(^{-1}))</th>
<th>Shear Eroded Filter Cake Thickness (inch)</th>
<th>Erosion time (hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ES01</td>
<td>90</td>
<td>20</td>
<td>0.045</td>
<td>1</td>
</tr>
<tr>
<td>ES02</td>
<td>89</td>
<td>200</td>
<td>0.040</td>
<td>1</td>
</tr>
</tbody>
</table>

Figure 4.6 shows that the shear eroded filter cake thickness data closely match with the un-eroded data line. The data do not show any effect of fluid erosion.
Figure 4.7 shows that the 200 s$^{-1}$ erosion rate had not affected the appearance of the filter cake. The filter cake has the same elasticity and thickness as the un-eroded filter cake.

Figure 4.7  Filter Cake after 200 s$^{-1}$ Shear Rate Erosion

Figure 4.8 shows that the filter cake was peeled away from the core sample without sticking or laceration. From these results, the filter cake thickness did not decrease under the 200 s$^{-1}$ shear rate condition. This result was repeated at the lower 20 s$^{-1}$ shear rate.
In this study, the filter cakes were built up by static method. Static method means the fracturing fluid did not flow during the filter cake build up. In contrast, the dynamic method builds up the filter cake when the fluid flows through the fracture at a certain shear rate. The dynamic method simulates the field situation more closely. Dynamic method requires special conditions of high pressure and high flow rate. The centrifugal and peistaltic pumps are not capable at this condition, so this study was conducted with the static method. Future experiments will improve to doing a dynamic method.

The dynamic method is expected to produce different results. The filter cake thickness is controlled by the shear stress, thus the filter cake is harder to build up with dynamic method. The shear stress impact on the filter cake is unpredictable.
4.4 Leakoff Coefficient

The following equation relates the leakoff coefficient to the leakoff volume and the leakoff time.

\[ V_L = 2AC_w \sqrt{t} \]  \hspace{1cm} (4.3)

Figure 4.9 shows a linear relationship of the square root of the leakoff volume versus leakoff time. The data points come from 11 experiments, in which the leakoff pressure was set at 500 psi and the polymer concentration was set at 40 lb/mgal. The core sample permeability in these experiments varied from 0.3 to 0.6 md.

Figure 4.9 Leakoff Volume versus Leakoff Time Correlation for Each Experiment

Table 4.3 shows the calculated leakoff coefficient for each experiment.
Table 4.3 Leakoff Coefficient

<table>
<thead>
<tr>
<th>Name</th>
<th>Leakoff Coefficient (ft/min$^{0.5}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>FS05</td>
<td>0.0011542</td>
</tr>
<tr>
<td>FS06T</td>
<td>0.0011020</td>
</tr>
<tr>
<td>FS06B</td>
<td>0.0013832</td>
</tr>
<tr>
<td>FS07</td>
<td>0.0012194</td>
</tr>
<tr>
<td>FS08</td>
<td>0.0012373</td>
</tr>
<tr>
<td>FS09</td>
<td>0.0010336</td>
</tr>
<tr>
<td>FS10</td>
<td>0.0010533</td>
</tr>
<tr>
<td>FS11</td>
<td>0.0010443</td>
</tr>
<tr>
<td>FS12</td>
<td>0.0012168</td>
</tr>
<tr>
<td>FS13</td>
<td>0.0012311</td>
</tr>
<tr>
<td>FS14</td>
<td>0.0013041</td>
</tr>
<tr>
<td>FS15</td>
<td>0.0013059</td>
</tr>
</tbody>
</table>

4.5 Filter Cake Thickness Coefficient

In Figure 4.5, the correlation between the total leakoff fluid volume and the total filter cake thickness applies in the hydraulic fracturing field. To apply this correlation in the field, the leakoff volume needs to be converted into the filter cake concentration factor. The concentration factor is defined as $F_{fc} = \frac{V_{fc} + V_L}{V_{fc}}$, where $V_{fc}$ is the filter cake volume and $V_L$ is the leakoff fluid volume. Figure 4.10 illustrated the definition of this factor.
The filter cake thickness prediction equation can be written as,

\[ T_{FC} = F_{Fe} C_{FCT} \]  \hspace{1cm} (4.4)

The Figure 4.5 is re-plotted as following Figure 4.11.
From Figure 4.11, the filter cake thickness empirical equation is $T_{FC} = 0.0803 \times F_{FC}$.

The unit of $C_{FCT}$ is millimeter.

The Filter Cake Thickness Coefficient $C_{FCT}$ depends on the polymer concentration in the fracturing fluid. A 40 lb/mgal polymer concentration was used in these experiments. A relationship of the polymer concentration $C_p$ and filter cake thickness coefficient can be written as

$$C_{FCT} = 0.0803 \frac{C_p}{40}$$

(4.5)

where $C_p$ is polymer concentration in $\frac{lb}{mgal}$. 

![Figure 4.11 Concentration Factor Correlation](image.png)
5. CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

The following conclusions are drawn from this study:

1. Filter cake was built up statically in the lab and the thickness was measured with two different methods.

2. The laser profilometer has been proven an effective tool to measure the filter cake thickness.

3. The data measured with the profilometer compared with micrometer measurement.

4. A crosslinked guar fluid correlation between the leakoff volume and the filter cake thickness was produced.

5. An experiment setup used to shear erode the filter cake is built up and tested.

6. No effect of filter cake erosion was observed at $200 \text{ s}^{-1}$ shear rate.

7. The leakoff coefficient is very stable in those experiments.

8. An equation was derived to predict the filter cake thickness based on the leakoff time, the leakoff coefficient and the filter cake concentration factor.

5.2 Future Work Recommendations

Measure the gel concentration in the filter cake and the remaining fracturing fluid.

Build up the filter cake dynamically. The fluid has to be circulated and pressurized.
Add proppant into the fracturing fluid when doing the filter cake erosion experiment to simulate the field condition better.

Place the proppant on the filter cake and measure the conductivity.

Put breaker into the fluid and measure the filter cake thickness after certain amount of time. The filter cake thickness and yield stress is expected to decrease proportional to breaker concentration and shut off time.

Future study can be designed to measure the filter cake thickness by laser and leakoff fluid by balance, thus can record the filter cake thickness and leakoff fluid in real time.
NOMENCLATURE

\( A \) = Cross-sectional area (in\(^2\))

\( C_W \) = Leakoff coefficient

\( C_{FCT} \) = Filter cake thickness coefficient (mm)

\( C_p \) = Polymer concentration (lb/mgal)

\( C_s \) = Surface concentration (lb/ft\(^2\))

\( F_{FC} \) = Fracturing fluid concentration factor

\( h \) = Fracture height (ft)

\( K' \) = Fluid rheology data

\( L \) = Length over pressure drop (in.)

\( M \) = mass/area of filter cake, m/L\(^2\), lbm/ft\(^2\)

\( M_{\text{core}} \) = Core weight, g

\( n' \) = Fluid rheology data

\( q \) = Fluid flow rate (L/min)

\( R \) = Universal gas constant (J/mol K)

\( t \) = Leakoff time (1/min)

\( v \) = Fluid flux (ft/min)

\( V_{fc} \) = Filter cake volume (gal)

\( V_L \) = Filtrated fluid volume (gal)

\( V_{\text{core}} \) = Core bulk volume

\( W \) = Mass flow rate (kg/min)

\( w \) = Fracture width (ft)
\( x_f \) = Fracture length (ft)

\( z \) = Compressibility factor of gas (Dimensionless)

\( \phi \) = Porosity

\( \rho_r \) = Core grain density (lbm/ft\(^3\))

\( \rho_w \) = Water density (lbm/ft\(^3\))

\( \mu \) = Fluid viscosity (cp)

\( \beta \) = Inertial flow coefficient (1/ft)

\( \tau_w \) = Shear stress of fluid at filter cake wall, m/L\(^2\), lbm/ft\(^2\)

\( \tau_y \) = Shear yield stress of filter cake wall, m/L\(^2\), lbm/ft\(^2\)

\( \tau_{yield} \) = Static yield stress of filter cake wall, m/L\(^2\), lbm/ft\(^2\)

\( \gamma_w \) = Shear rate on the filter cake wall, 1/T, 1/s
REFERENCES


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