UNCONVENTIONAL RESERVOIR ENGINEERING PROJECT

COLORADO SCHOOL OF MINES

Research Summary

# MEASURING MASS OF GAS IN-PLACE IN CORES

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# Outline

- ❑ Objective
- ❑ Background
- ❑ Summary of Previous Work
- ❑ Work in Progress
- ❑ Future work

# Objective

- Measure mass of gas in-place (GIP) in cores (not crushed)
  - Develop a method that determines mass by measuring the frequency of oscillation
  - Test the method on Berea sandstone cores
  - Verify that mass of GIP can be measured using the oscillation method
- Measure Gas in-Place (GIP) in other tighter rocks

# Background

$$m = m_0 - m_a + m_p + \Delta m$$

| | | |
|---|---|---|
| $m$ | = | Effective Mass from Oscillation (g) |
| $m_0$ | = | Mass of sample at ambient condition (g) |
| $m_a$ | = | Added Mass due to co-acceleration of gas external to the sample (g) |
| $m_p$ | = | Mass due to co-acceleration of gas inside pores of the sample (g) |
| $\Delta m$ | = | Mass attributed to gas condensation and adsorption (g) |

# Summary of Previous Work

$$GIP = \Delta m + m_p = m - m_0 - m_a$$

$$m = \frac{k}{4\pi^2 f^2}$$

$$m_a = \alpha P$$

m = Effective mass from Oscillation (g)
f = Frequency from Oscillation (Hz)
k = Spring constant (N/mm)

α = Added mass coefficient (g/psi)
P = Pressure (psi)

# Summary of Previous Work

## Conventional Rocks

$$\boldsymbol{GIP} = m_p = \left(\frac{\boldsymbol{k}}{\boldsymbol{4\pi^2 f^2}}\right) - m_0 - (\boldsymbol{\alpha P})$$

$m_0$ = Mass of sample at ambient condition (g)

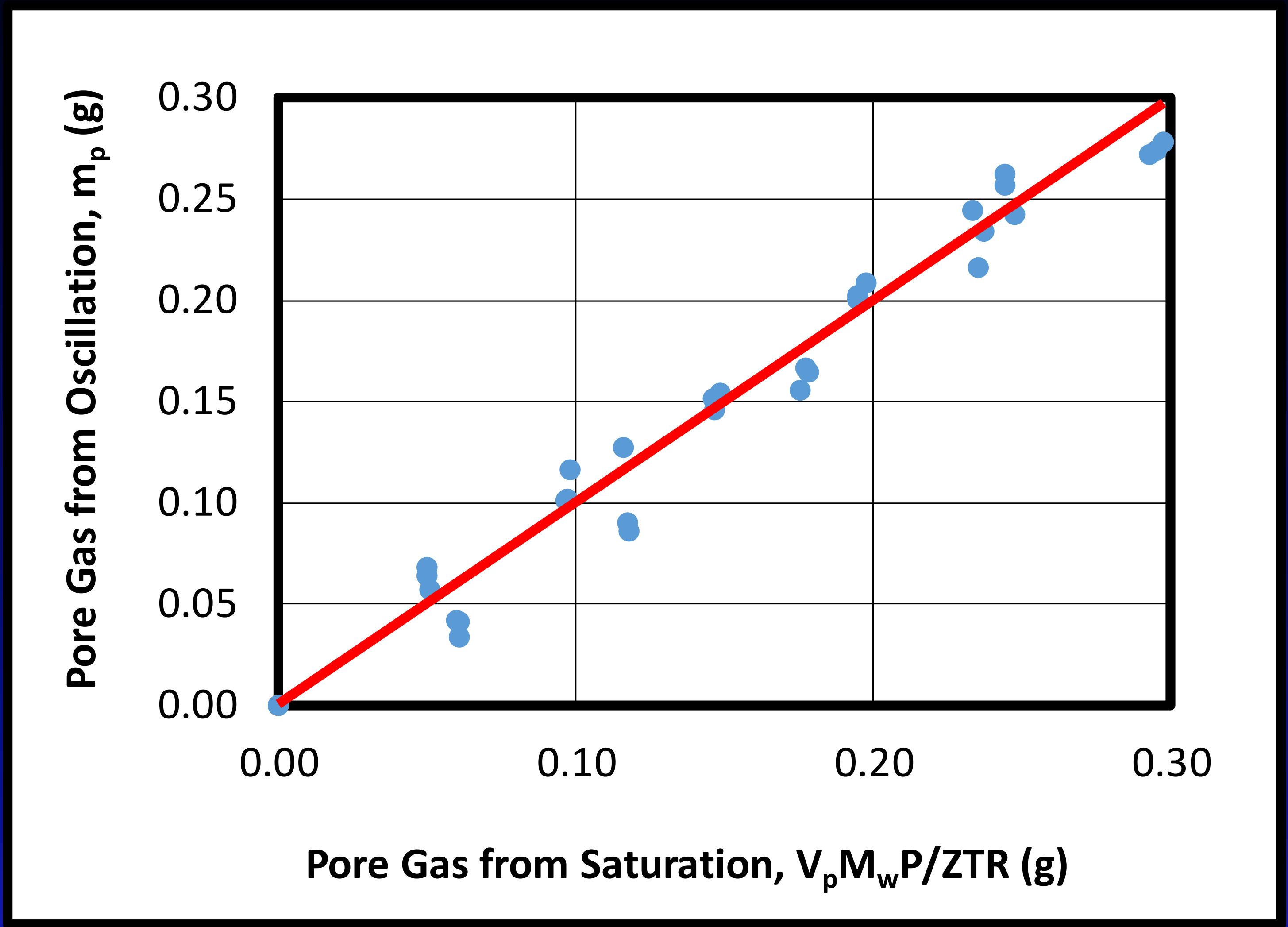
f = Frequency from Oscillation (Hz)

k = Spring constant (N/mm)

α = Added mass coefficient (g/psi)

P = Pressure (psi)

# Summary of Previous Work



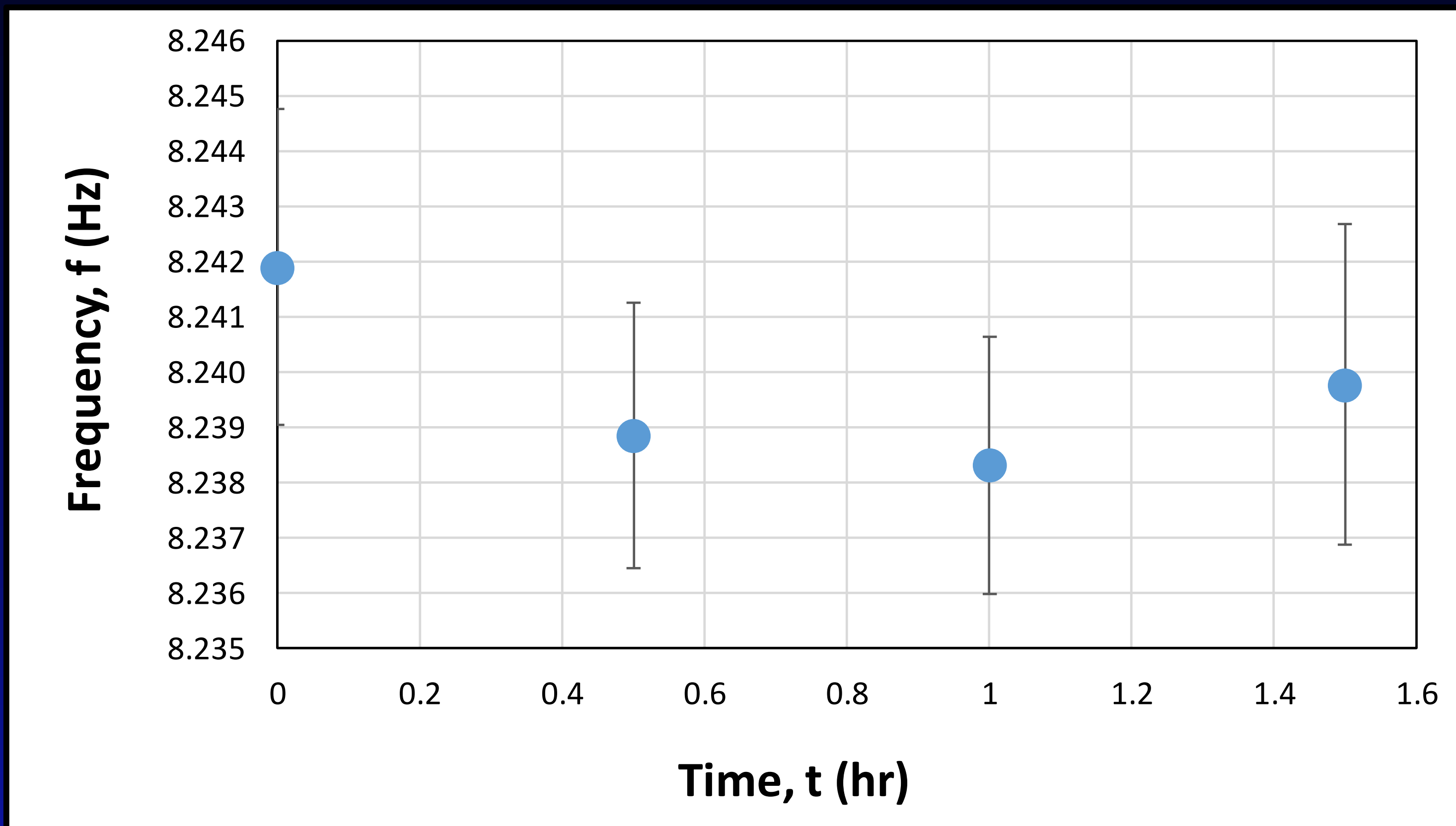
# Work in Progress

## Apply The Oscillation Method on Unconventional Cores

- Using same procedures as Conventional Cores
- Gas-in-place (GIP) includes both Free Gas Mass ($m_p$) and Mass attributed to gas condensation and adsorption($\Delta m$)
- Long transient flow → long injecting time
    - Mass increases with time
    - Frequency decreases with time
- Unfortunately, detect no changes in frequency (Hz)

# Work in Progress

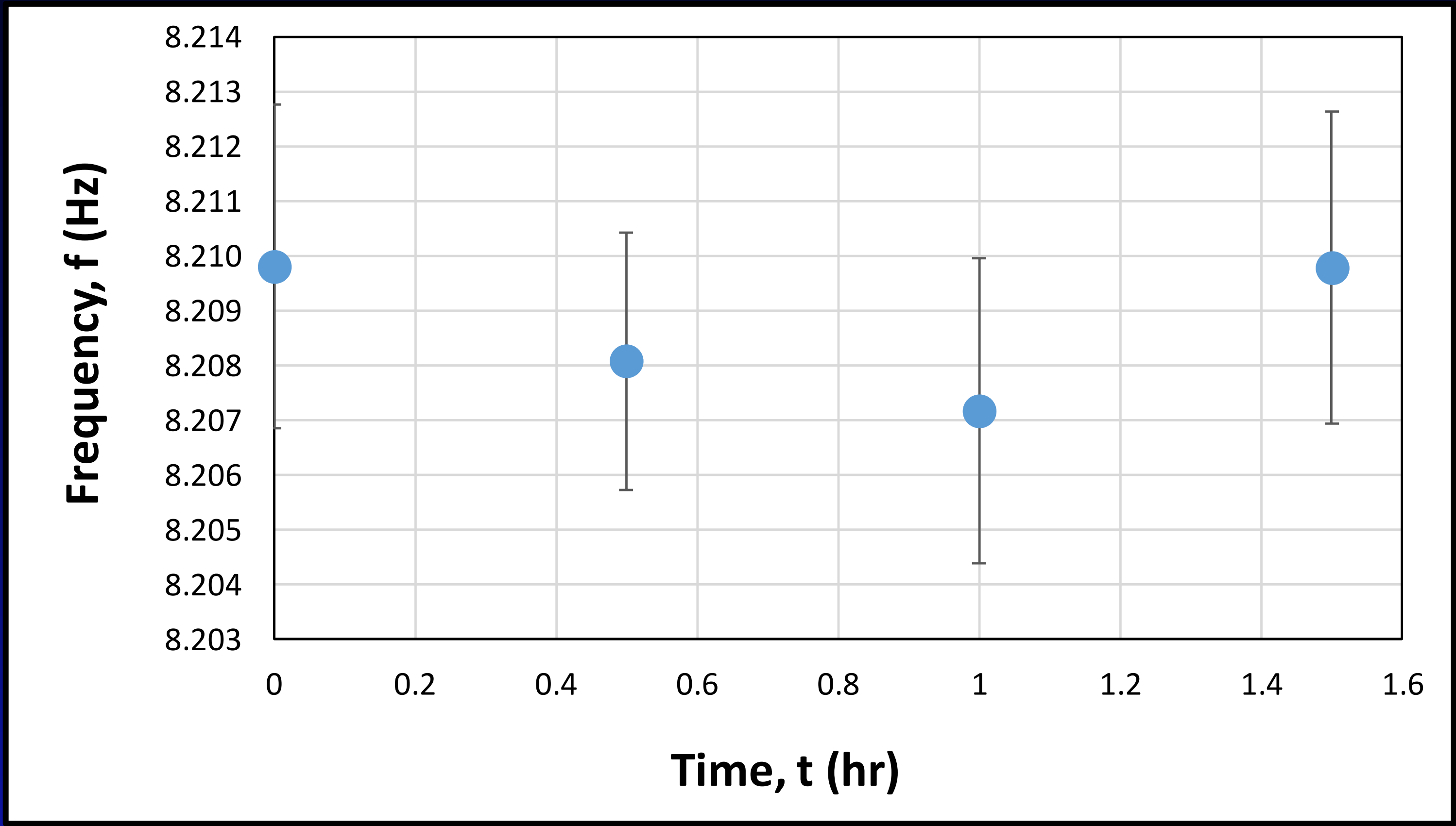
Pressure = 230 psia




*** Every point in Figure above represents an average of 30 measurements**

# Work in Progress

Pressure = 300 psia




* Every point in Figure above represents an average of 30 measurements

# Future Work

- Replace the old vacuum pump with the better pump
- Replace the current frequency counter, oscilloscope with the better frequency measurement tools
- Inject Gas with higher pressure to increase the mass of gas
- Expect to see changes in frequency with time
- Validate that the oscillation method is applicable for measuring gas-in-place (GIP) in Unconventional rocks

# RESEARCH SUMMARY

## Using Oscillations to Determine Capillary Condensation in MCM-41

**Keerthana Krishnan, Xiaolong Yin**
**Collaborator: Dr. Brian Trewyn**

# Outline

- ❑ Objective & Motivation
- ❑ Background
- ❑ Materials & Equipment
- ❑ Methodology
- ❑ Results
- ❑ Conclusion
- ❑ Future work

# Objective & Motivation

❖ Objective

- Measure capillary condensation in artificially created MCM-41 nanosilica material with specific pore size using the new oscillation based-gravimetric method

❖ Motivation

- Condensation of hydrocarbon fluids in micropores and mesopores in unconventional resources is important as nanopores contribute to a significant amount of porosity in the unconventional resources

# Background

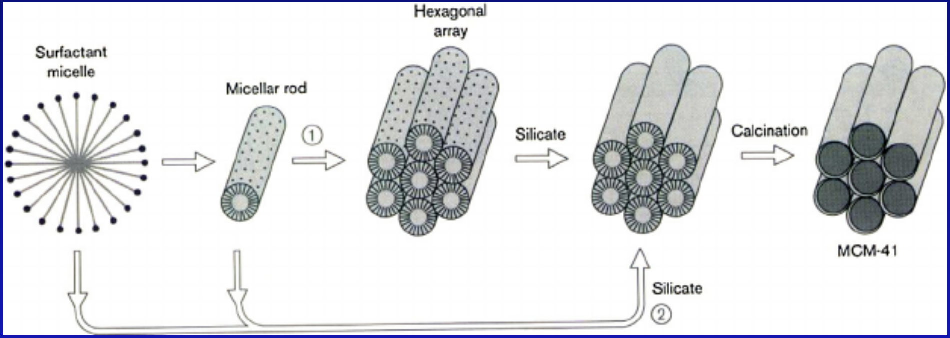
$$\Delta m = m - (m_0 - m_a + m_p)$$

| | | |
|---|---|---|
| $m$ | = | Effective Mass from Oscillation (g) |
| $m_0$ | = | Mass of sample at ambient condition (g) |
| $m_a$ | = | Added Mass due to co-acceleration of gas external to the sample (g) |
| $m_p$ | = | Mass due to co-acceleration of gas inside pores of the sample (g) |
| $\Delta m$ | = | Mass attributed to gas condensation and adsorption (g) |

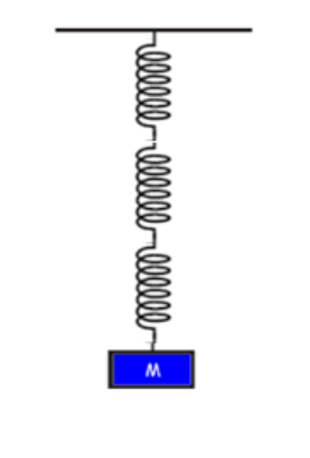
# Materials & Equipment

## Mobile Crystalline Material (MCM-41)

- Contains Mesopores
- Typically synthesized by silica-surfactants mixtures
- Porosity can be as high as 80%
- Pore diameter typically tuned to:  2-10 nm
- High pore volume, high BET surface area



# Materials & Equipment



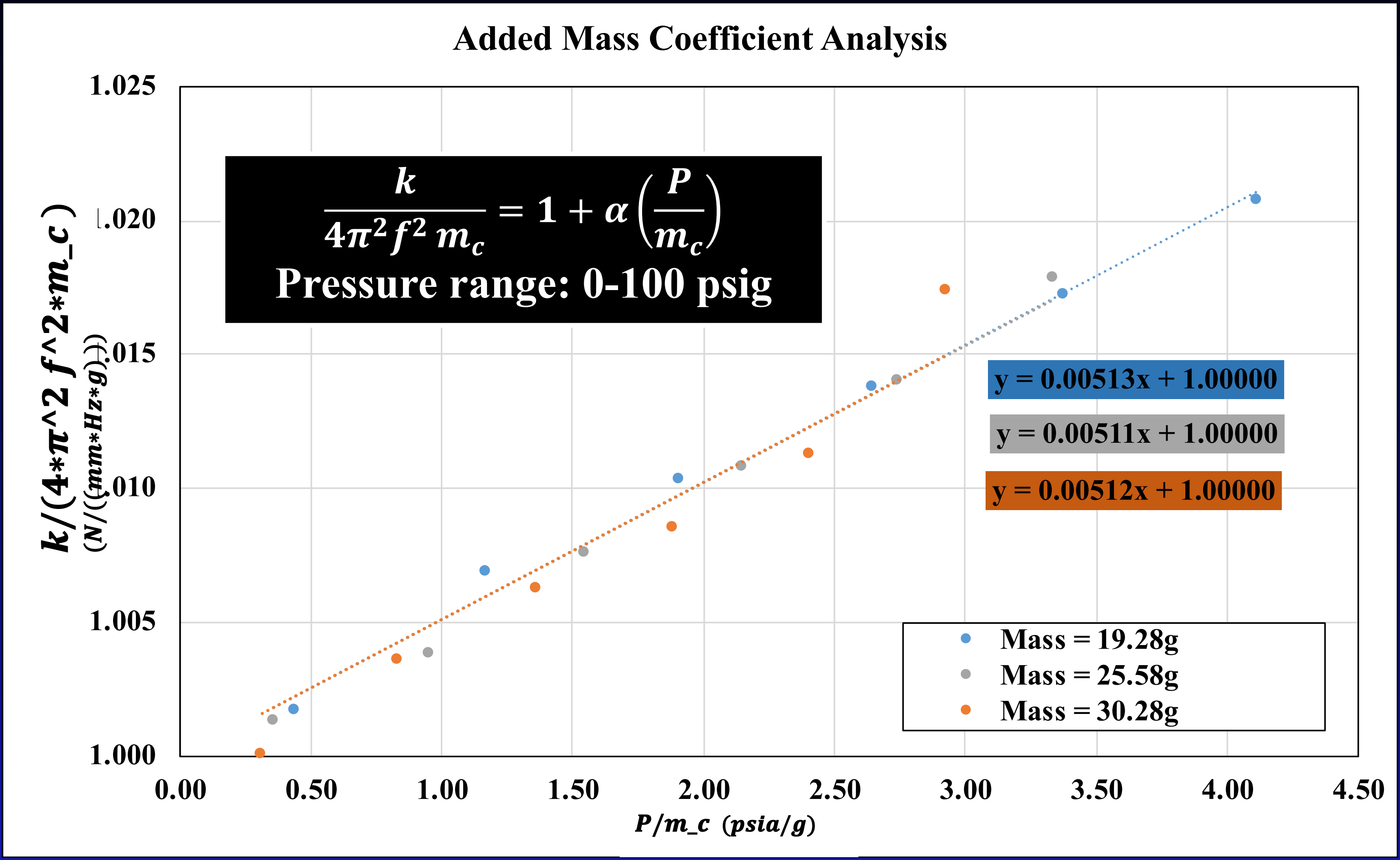

- To account for small mass of sample, springs had to be connected in series and the total weight ( test object, weight holder and magnet) had to reduced

- To reduce the deadweight from the weight holder
- The sample was filled in the weight holder by compressing it until we reached 10.000 g

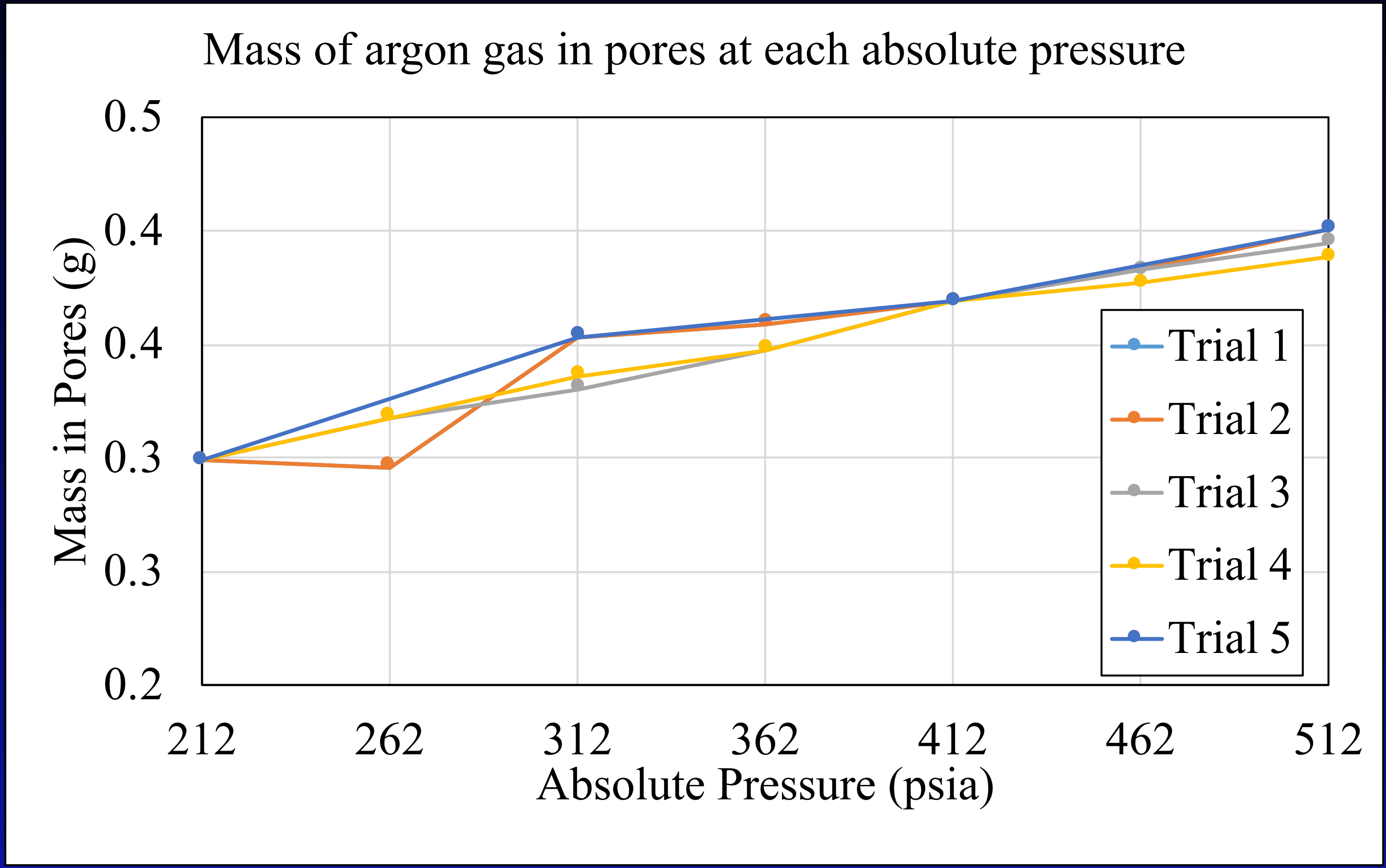
# Methodology

1. Determine Added Mass ($m_a$)
   - Using Aluminum bars (Non-porous media) with the same cross sectional area as the weight holder.
2. Determine Pore Gas Mass ($m_p$)
   - Using Argon Gas (Non-Adsorbing, Non-Condensing Gas)
3. Determine Mass due to Capillary Condensation ($\Delta m$)
   - Using High Purity Propane Gas (99.999%) (Condensing Gas)

# Results


Added Mass Coefficient Analysis
$$\frac{k}{4\pi^2 f^2 m_c} = 1 + \alpha\left(\frac{P}{m_c}\right)$$
Pressure range: 0-100 psig
y = 0.00513x + 1.00000
y = 0.00511x + 1.00000
y = 0.00512x + 1.00000
Mass = 19.28g
Mass = 25.58g
Mass = 30.28g
1.025
1.020
1.015
1.010
1.005
1.000
0.00
0.50
1.00
1.50
2.00
2.50
3.00
3.50
4.00
4.50
P/m_c (psia/g)
k/(4*π^2 f^2*m_c) (N/((mm*Hz*g)))

# Results

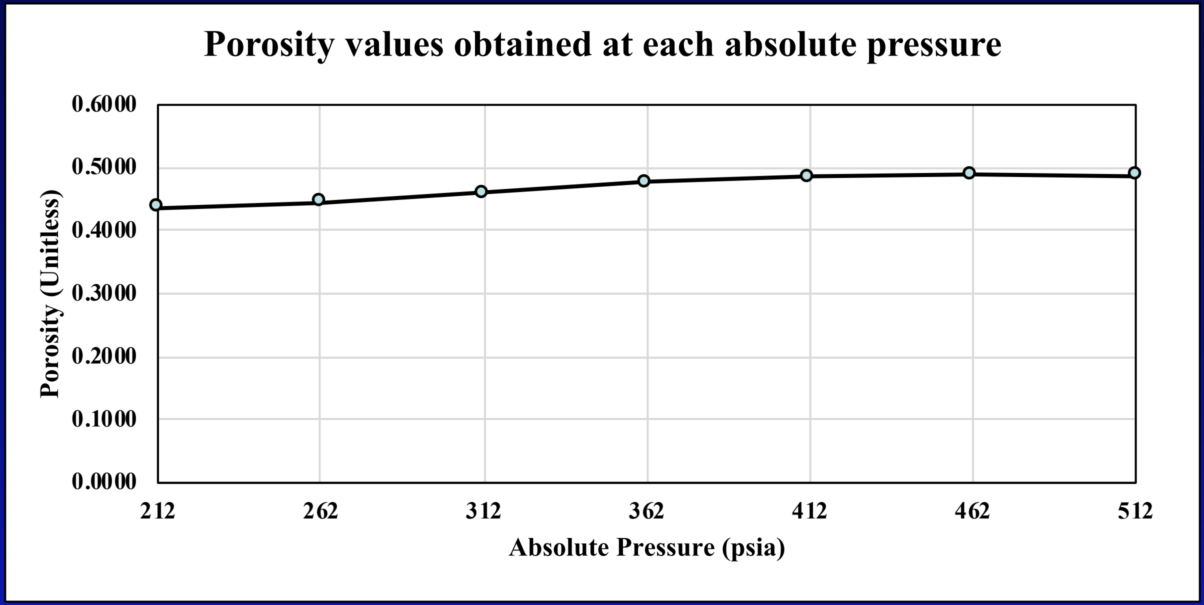


Mass of argon gas in pores at each absolute pressure
0.5
0.4
0.4
0.3
0.3
0.2
Mass in Pores (g)
212
262
312
362
412
462
512
Absolute Pressure (psia)
Trial 1
Trial 2
Trial 3
Trial 4
Trial 5

# Results

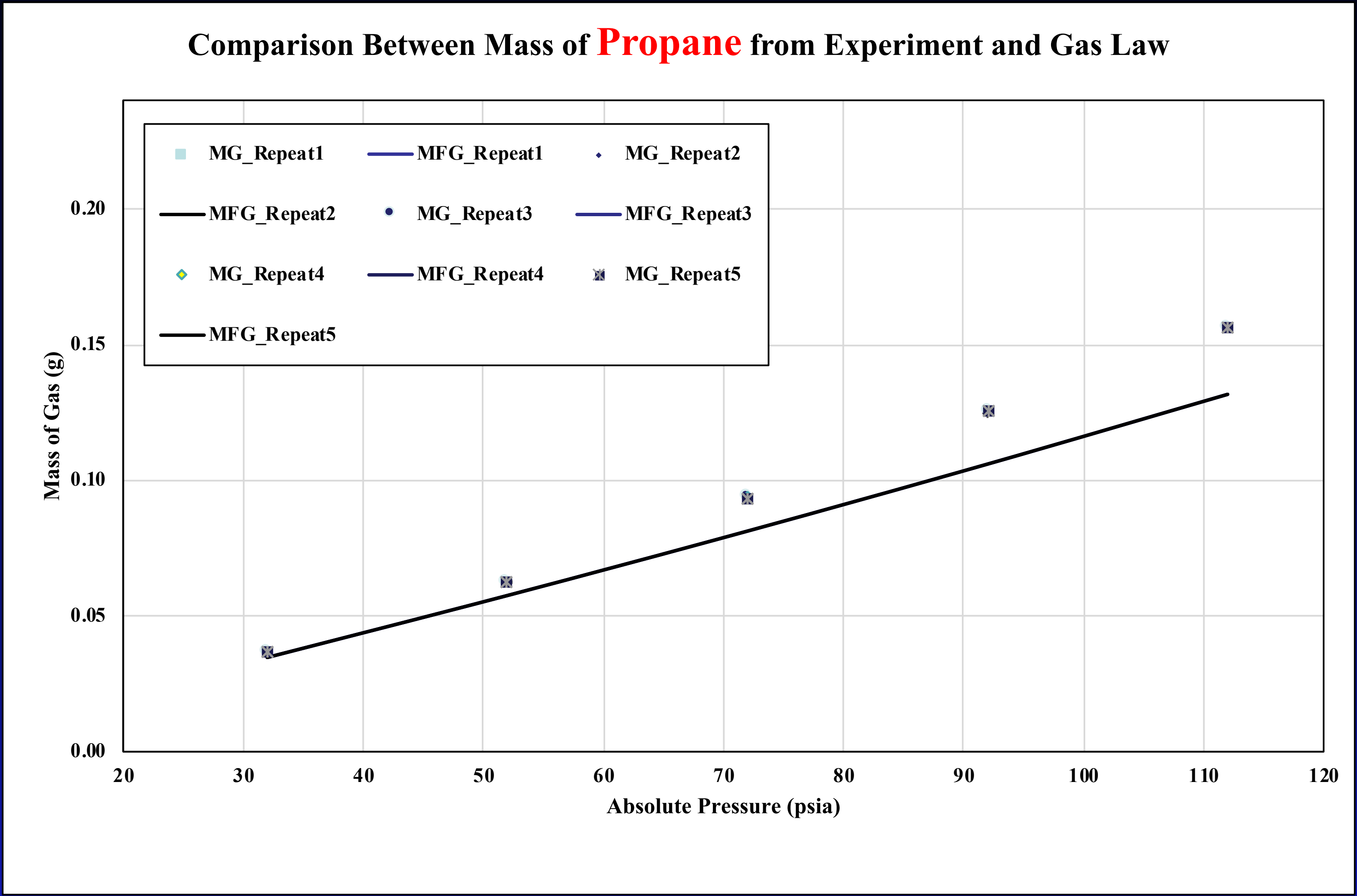
$$\text{Pore Volume (cc)} = \frac{\text{Mass in pores (g)}}{\text{Density of Argon (Using absolute Pressure)} \left(\frac{g}{cc}\right)}$$

$$\text{Porosity} = \frac{\text{Pore volume (cc)}}{\text{Bulk volume (cc)}}$$




**Pore volume = 8.00 cc**
**Porosity = 46.9 %**

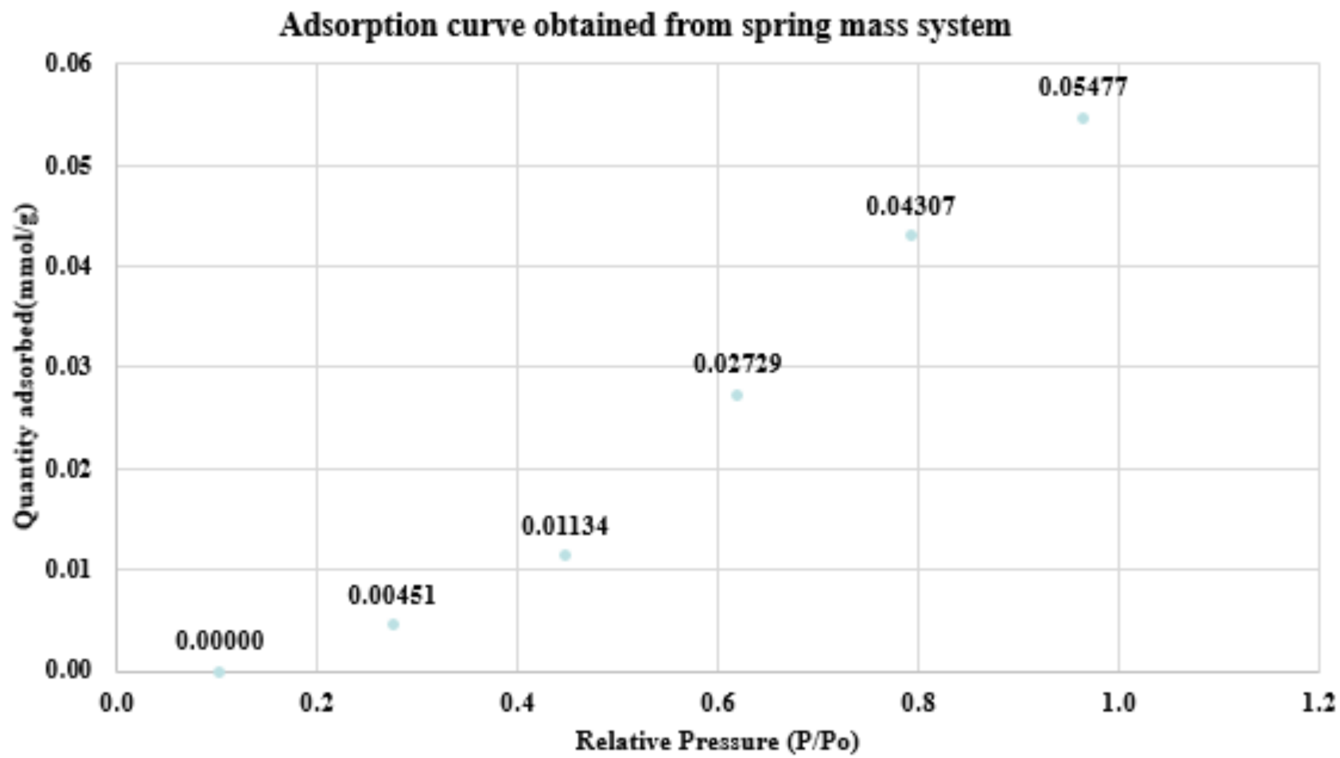
# Results



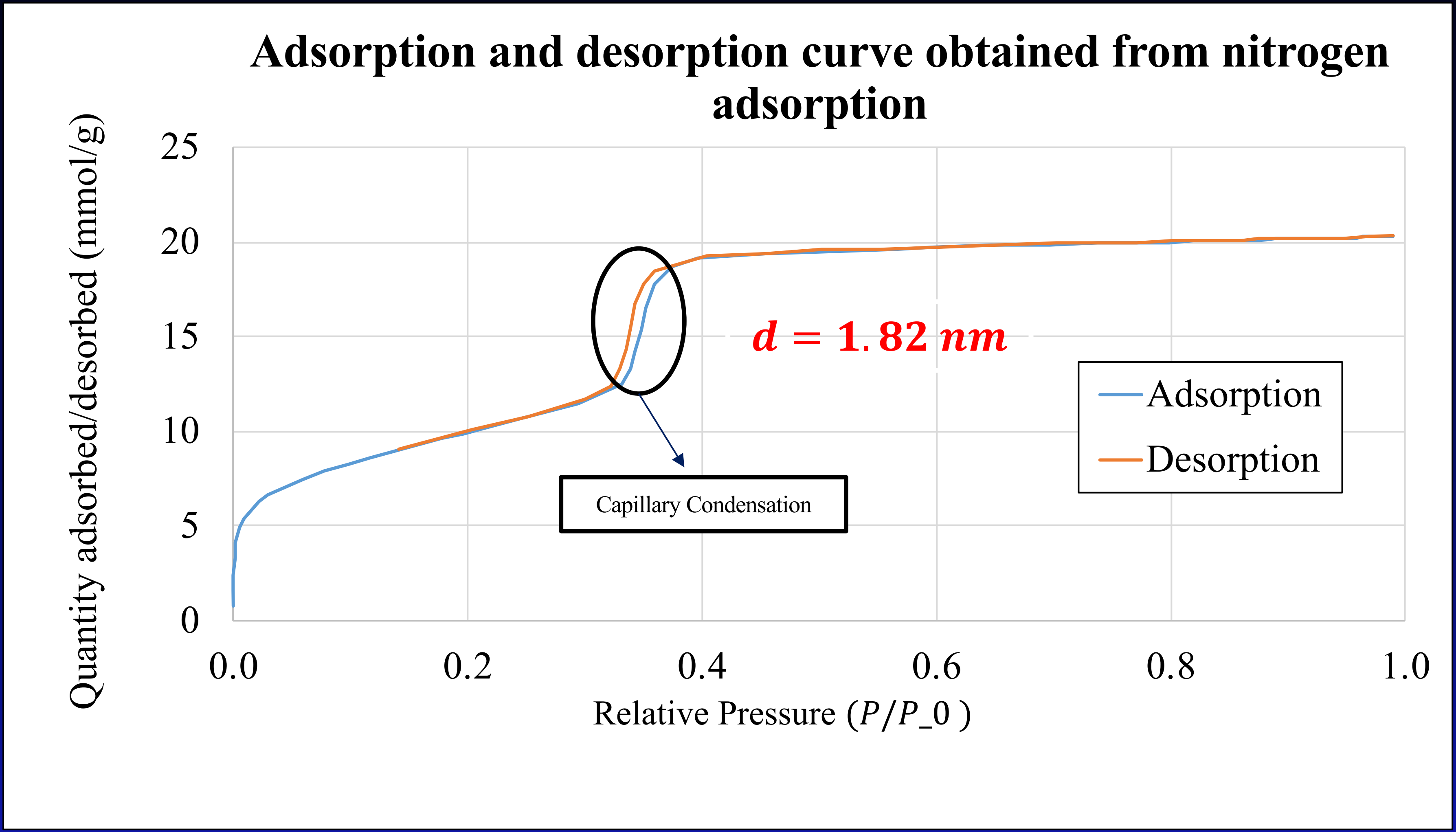
Comparison Between Mass of Propane from Experiment and Gas Law
MG_Repeat1
MFG_Repeat1
MG_Repeat2
MFG_Repeat2
MG_Repeat3
MFG_Repeat3
MG_Repeat4
MFG_Repeat4
MG_Repeat5
MFG_Repeat5
Mass of Gas (g)
0.00
0.05
0.10
0.15
0.20
20
30
40
50
60
70
80
90
100
110
120
Absolute Pressure (psia)

# Results

## Quantity adsorbed graph from spring-mass system



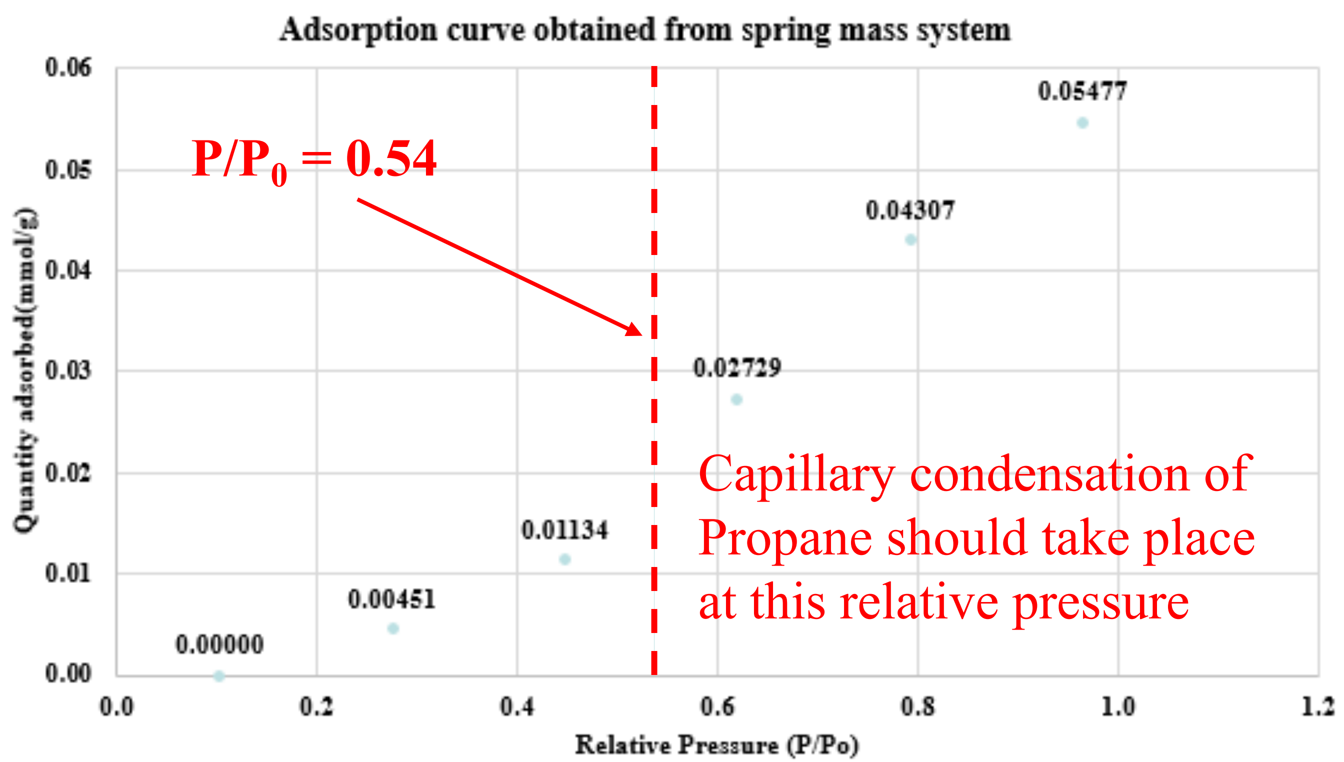
# Results



Adsorption and desorption curve obtained from nitrogen adsorption
Quantity adsorbed/desorbed (mmol/g)
25
20
15
10
5
0
0.0
0.2
0.4
0.6
0.8
1.0
Relative Pressure $(P/P\_0)$
$d = 1.82\ nm$
Capillary Condensation
Adsorption
Desorption

# Results

## Quantity adsorbed graph from spring-mass system



# Conclusion

- The oscillation method successfully detected additional gas-in-place from Propane – Potentially due to condensation
- We did not observe a rapid increase in mass at the location where we expect capillary condensation
- Sensitivity and procedure of the experiment need to be improved in the future – The objective is to generate data comparable to Nitrogen isotherm

# Future Work

## Replace Old Equipment

- Replace the old vacuum pump with the better pump
- Replace the current frequency counter, oscilloscope with the better frequency measurement tools

# Q&A