

Testing Method for True Density of Ceramic Fracturing Proppants

1. Background

Fracturing proppants, also known as ceramic particles or ceramic sand, are sintered from high-quality bauxite and other raw materials. These proppants are widely used in deep-well and high-pressure oil/gas reservoir fracturing operations.

During deep oil and gas well extraction, hydraulic fracturing is applied to low-permeability reservoirs under high closure pressure. This process fractures the hydrocarbon-bearing rock layers, creating channels for oil and gas flow. Proppants are injected with high-pressure fluid into fractures to prevent closure under stress, maintaining high conductivity and enhancing production. Field data demonstrates that ceramic proppants can increase well productivity and extend operational lifespan.⁽¹⁾

True density is defined as powder mass divided by the **true volume** of the powder, which is the powder volume excluding any internal pores, cracks, or voids. If the material is porous, like ceramic particles, the true density can differ significantly from the bulk density (pore volumes not subtracted). The true density of ceramic particles is directly related to proppant effectiveness. The particles sintered from bauxite tend to have high true densities, which allow them to resist crushing and breakage. However, some applications require lightweight proppants which resist particle settling and are typically less costly.⁽¹⁾ Therefore, accurate true density measurements are necessary to improve these technologies.

2. Methods of True Density Testing

The **Densi 100 True Density Analyzer** from AMI measures true density by employing the gas displacement method. As demonstrated in the scientific literature, helium is recommended as the displacement gas for true density measurements due to its small molecular radius, high diffusivity, and chemical inertness.⁽²⁾ These properties allow helium to penetrate fine pores and cracks of powder materials.

By measuring pressure changes in a calibrated chamber, the **true volume** and **true density** of the sample are determined. The procedure is as follows:

Step 1: Empty Sample Cup Calibration

- ✓ Record initial pressure ($P_{0,empty}$) of sample and reference chamber, charging pressure ($P_{ref,empty}$) of reference chamber, and equilibrium pressure ($P_{eq,empty}$). Calculate the calibration parameter $M_{P,empty}$ using Equation (1):

$$M_{P,empty} = \frac{P_{eq,empty} - P_{0,empty}}{P_{ref,empty} - P_{eq,empty}} \quad (1)$$

Definitions:

- ✓ Initial pressure (P_0) – pressure inside sample chamber before He is introduced
- ✓ Charging pressure (P_{ref}) – pressure of helium in reference chamber before opening to the sample chamber
- ✓ Equilibrium pressure (P_{eq}) – pressure measured after sample chamber is opened to reference chamber

Step 2: Sample Measurement

- ✓ Load the sample and record $P_{0,sample}$, $P_{eq,sample}$, and $P_{ref,sample}$. Calculate $M_{P,sample}$ using Equation (2):

$$M_{P,sample} = \frac{P_{eq,sample} - P_{0,sample}}{P_{ref,sample} - P_{eq,sample}} \quad (2)$$

Step 3: True Volume Calculation

- ✓ Determine the true volume (V_T) using Equation (3), where V_d is the chamber volume:

$$V_T = V_d \times (M_{P,empty} - M_{P,sample}) \quad (3)$$

Step 4: True Density Calculation

- ✓ Compute true density (D_T) via Equation (4).

$$D_T = \frac{\text{Mass of sample}}{V_T} \quad (4)$$

The schematic of the Densi100 is shown in Figure 1, which shows the sample chamber, reference chamber, pressure values, and connecting pipelines.

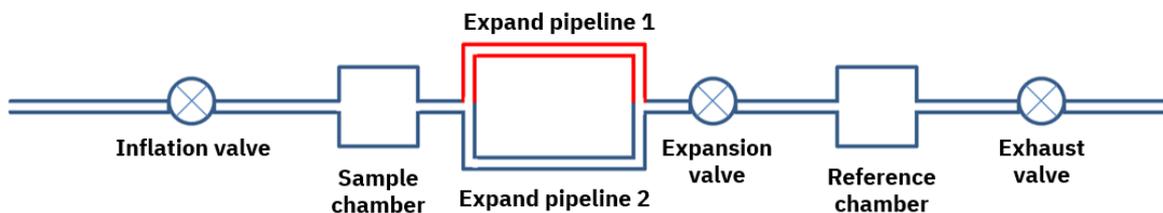


Figure 1: Instrument schematic of Densi100 True Density Analyzer from AMI

2. Results for ceramic proppant samples

| Sample | True Density (g/cm ³) | Relative Error (%) | Bulk Density (g/cm ³) | Difference between True Density and Bulk Density |
|--------|-----------------------------------|--------------------|-----------------------------------|--|
| 1 | 2.8605 | 0.08 | 2.77 | 0.0905 |
| 2 | 2.8493 | 0.05 | 2.79 | 0.0593 |
| 3 | 3.0875 | 0.04 | 3.03 | 0.0575 |
| 4 | 3.0657 | 0.1 | 3.05 | 0.0157 |

Table 1: True density test results of fracturing proppants

The Densi100 produced reliable, precise true density measurements of ceramic proppants with low relative error values, shown in Table 1. These results were significantly higher compared to the bulk density measurement, which illustrates the difference between the two parameters.

| Model | Densi 100 |
|--------------------|--|
| Principle | Gas displacement method |
| Pretreatment | Gas purge, Flow |
| Pressure | 0-150 kPa (Gauge) |
| Accuracy | 0.03% |
| Repeatability | 0.02% |
| Cell Volume | Nominal: 100 ml or 10 ml Available Inserts: 35 ml, 10 ml or 3.5 ml, 1 ml |
| Calibration method | Automatic calibration with calibration blocks |
| Gases | He (N ₂) |
| Testing Range | 0.0001 g/cm ³ to the infinity; |
| Volume and Weight | L 15.0 in (380 mm) × W 11.0 in (280 mm) × H 11.0 in (280 mm), 22 lbs (10 kg) |
| Power Requirements | 100 or 240 VAC, 50/60 Hz, maximum power 100 W |

Table 2: Instrument specifications and capabilities of the Densi100 True Density Analyzer from AMI



Figure 2: Densi100 True Density Analyzer from AMI

4. Conclusions

The **Densi 100** True Density Analyzer delivers high precision ($\pm 0.03\%$) and reproducibility ($\pm 0.02\%$), fulfilling the rigorous requirements for absolute density testing of ceramic proppants. This method ensures reliable quality control in hydraulic fracturing applications.

5. References

- (1) Liang, F.; Sayed, M.; Al-Muntasheri, G. A.; Chang, F. F.; Li, L. A comprehensive review on proppant technologies. *Petroleum*, **2016**, *2*, 26-39.
- (2) Nguyen, H. G. T.; Horn, J. C.; Bleakney, M.; Siderius, D. W.; Espinal, L. Understanding material characteristics through signature traits from helium pycnometry. *Langmuir*, **2019**, *35*, 2115-2122.