Solid Density of Building Materials: Assessing Purity of Powders and Open Porosity of Concrete Components

Relevant for: building, bitumen, cement, purity, porosity, pycnometry, tapped density

The density of building materials such as cement and bitumen plays a significant role in production and performance. Purity of powders and the open porosity of cured pieces are important material characteristics that can be assessed from solid density measurements. Tapped density via the Autotap and skeletal density via the Ultrapyc 5000 give extremely accurate volume measurements that are required for the building industry and can be correlated to purity, porosity, and flow.

1 Introduction

Solid density is an important property of building materials that is used to control material quality from the raw powders to the formed final product. Geometric and skeletal densities enable researchers and producers to calculate the open porosity of a solid, cured sample, which leads to understanding critical properties such as strength, temperature tolerance, and “processability” [1], as, for example, some porosity is necessary to prevent fracturing during compression. Measuring skeletal density using the Ultrapyc series and bulk density using the Autotap provides information about important building material properties such as powder purity, density, and slab porosity.

For example, the density of bitumen (the major component within asphalt) plays a crucial role in making roads, but measuring this property has traditionally been a messy and lengthy process. Gas pycnometry using the Anton Paar Ultrapyc series can make this process simple, clean, and quick. Samples with bulk volumes as large as 135 cm^3 can be accommodated (thereby improving sampling statistics), purged of air and run multiple times automatically, and a printed report generated within minutes. The sample is recovered unharmed and dry, and the very same aliquot can be used for subsequent analyses or other tests. This method has already been adopted by a number of cement companies worldwide, and is standard in applications such as petroleum coke, pitch, coatings, carbon, cellular plastics, soils, ceramics, and catalysts, among others.

2 Building Material Purity

The Ultrapyc 5000 was used to measure the skeletal density of commercially-available cement patcher and dolomite. Deviations from the theoretical density of quartz (2.67 g/cm^3), which is the main ingredient in the cement patcher, and dolomite (2.85 g/cm^3) can indicate different impurities are present in the samples. Because these are fine powders, the *PowderProtect* mode of the Ultrapyc 5000 was utilized to expand gas from the reference chamber to the sample chamber to prevent elutriation of the powder. This allows high target pressures to be utilized to obtain the most accurate data possible. Sample measurement parameters are given in Table 1 and the resulting skeletal density measurements are shown in Table 2. Excellent repeatability was observed.

It was found that the dolomite studied was, within error, 100% pure. The purity of the cement was more difficult to definitively determine because of the mixture of components. For example, the measured skeletal density is larger than the density of pure quartz, indicating the impurities are higher density components such as quicklime (CaO).

Parameter	Setting
Cell size	Large
Gas type	Helium
Target pressure	10 psig
Flow direction mode	Reference first
Equilibration	Pressure
Preparation mode	Flow, 1 minute
Maximum runs	15
Runs to average	3

Table 1: Ultrapyc 5000 measurement parameters

Sample	Density (g/cm^3)				Repeatability (%)
	Run 1	Run 2	Run 3	Average	
Concrete	2.7490	2.7481	2.7477	2.7482	0.02
Dolomite	2.8538	2.8517	2.8536	2.8530	0.03

Table 2: Uncured density measurements

3 Tapped Density

Some materials are used as lubricants in the manufacturing process. The flowability of these lubricants can be described using tapped density to obtain the Hausner ratio (HR) and Compressibility Index (CI) [2], described in the equations below.

$$HR = \frac{V_0}{V_f}$$

$$CI = \frac{V_0 - V_f}{V_f} * 100$$

Where V_0 is the initial volume before it is tapped, and V_f is the final volume after it no longer compresses.

The tapped density of 136.07 g of the commercially available cement patcher was measured using the Autotap. The results for this sample are shown in Table 3 and a HR of 1.42 and CI of 29.7 were calculated. These values indicate the cement patcher would exhibit poor flow [3].

Taps	Volume (cm^3)	Density (g/cm^3)
0	$V_0 = 96$	1.41
2000	$V_f = 67.5$	2.01

Table 3: Tapped density measurements

4 Bitumen Density

Knowing the density of bitumen/asphalt is essential in order to convert the volume to mass when selling the product. The density value is also required to categorize the asphalt/bitumen by density after distillation and when developing new and stronger products [4]. Previously, following ASTM D70 [5] these measurements were time consuming and messy. The Ultrapyc now makes the process quick, simple, and clean.

The Ultrapyc 5000 was used to measure the skeletal density of a commercially-available bitumen substitute, an asphalt filler, at 25 °C.

In order to efficiently and cleanly measure the density of the bitumen substitute, a disposable aluminum cup was used to hold the sample inside the sample cell as illustrated in Figure 1. This disposable cup eliminates the need to clean the instrument sample cell after measurements.

Figure 1: Disposable aluminum cup next to (a.) and inside of (b.) the small sample cell. The prepared sample cell is shown in (c.).

First, the volume of the empty disposable cup was measured. It was then filled with the bitumen substitute and carefully placed into the cell. The bitumen substitute was run *reference first* to minimize the effect of any vapor pressure and ensure no material contaminated the instrument. The entire process including: filling the cup with sample, entering the run parameters, performing the measurement, and preparing the instrument for the next measurement (by disposing of the disposable cup/sample) took under 30 minutes. Sample measurement parameters are given in Table 4 and the resulting skeletal density measurements are shown in Table 5. It is important to note that the output data is calculated with both the volume of the sample + the disposable cup. Thus, the volume of the disposable cup, determined previously, must be manually subtracted and the density recalculated with this new volume. As an alternative, the initial calibrations could also have been done with the cup before filling it with the sample.

Parameter	Setting
Cell size	Small
Gas type	Helium
Target pressure	10 psig
Flow direction mode	Reference first
Equilibration	Fixed time, 0.33 min
Preparation mode	Flow, 1 minute
Maximum runs	15
Runs to average	3

Table 4: Ultrapyc 5000 measurement parameters

Sample	Volume (cm^3)				Density (g/cm^3)	Repeatability (%)
	Run 1	Run 2	Run 3	Avg	Avg	
Bitumen substitute + Al cup	6.953	6.946	6.942	6.947	1.8010	0.07
Al cup	0.753	0.750	0.754	0.7523	2.7253	0.19
Bitumen substitute	6.200	6.196	6.188	6.195	1.6888	

Table 5: Bitumen substitute density measurements

5 Open Porosity of Cement Pieces

Slab strength and dissolution properties can be assessed from calculations of percent open porosity from skeletal density measurements. To calculate percent porosity, the geometric density is also needed. In this case, geometric or bulk density was obtained by dividing the measured mass by the calculated geometric volume of the cylindrical sample.

The cement and bitumen substitute samples were prepared as instructed on the commercial packaging and allowed to cure. The solid, cured pieces were then measured on the Ultrapyc 5000. The Ultrapyc 5000 was temperature controlled to 25 °C. The measurement parameters are shown in Table 6 and the resulting skeletal density measurements are shown in Table 7. Good repeatability between runs was observed. Coupled with the bulk volume, the open porosity of the cement and bitumen substitute pieces were calculated using the equation below to be 31.3%, and 37.6%, respectively.

$$\%\,porosity = \frac{V_B - V_S}{V_B} * 100$$

In this equation V_B is the bulk volume and V_S is the skeletal volume.

Parameter	Setting
Cell size	Medium
Gas type	Helium
Target pressure	18 psig
Flow direction mode	Sample first
Equilibration	Pressure
Preparation mode	Flow, 1 minute
Maximum runs	15
Runs to average	3

Table 6: Ultrapyc 5000 measurement parameters

Sample	Density (g/cm^3)				Repeatability (%)
	Run 1	Run 2	Run 3	Avg	
Cement	2.5778	2.5769	2.5765	2.5770	0.02
Bitumen substitute	1.9407	1.9403	1.9404	1.9404	0.008

Table 7: Cured sample density measurements

6 Conclusions

The Ultrapyc 5000 is ideal for measuring the density of building materials. Highly accurate and repeatable measurements ensure that skeletal densities can be measured easily and confidently and correlated with purity, as discussed in the dolomite analysis, and porosity properties as seen in the cured cement and bitumen-substitute analyses. Accurate skeletal density measurements allow researchers to quickly assess this material property and screen new materials. Additionally, gas pycnometry allows for a faster, cleaner, more accurate way to measure the density of bitumen as compared to more traditional methods, such as ASTM D70 and EN 15326.

7 References

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