Design and test of porosity testing apparatus for granular materials

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Abstract. In order to improve the accuracy and reliability of porosity measurement of granular materials, on the basis of equation of state of ideal gas and gas displacement method, a rapid measurement apparatus for porosity of granular materials with constant volume was studied and an automatic detection control system was developed. Within measurement range of 0.1~0.4 MPa, the standard stainless-steel spheres, plastic quadrangular prisms, cobblestones and fine gravels with different shapes accurately measurable were adopted, and it was found that the higher the inflation pressure of the system, the smaller the measurement error and the better the repeatability. Using standard stainless-steel sphere and cylinder with porosity of 16.02%, 25.14%, 35.57%, 49.05%, 58.42%, 67.79%, 76.57%, 85.36% and 94.14% respectively, the formula of compensation coefficient was given. The measurement error was 0.3%, which proved the reliability of the measurement results.

1. Introduction

Porosity is one of the basic physical characteristic parameters of materials in the process of filling, transportation, storage, drying, etc. It is also an indispensable basic original data and important engineering parameter in engineering design, equipment process evaluation and product analysis [1-4]. There are many kinds of granular materials and great differences in material configuration, especially non-uniform granular agricultural materials. At present, measuring the porosity of granular materials quickly and reliably under different working conditions is one of the weak points in the field of physical property measurement.

There are two methods to determine the porosity of materials: liquid displacement method and gas displacement method. The liquid replacement method is to use liquids that do not react with materials, such as water, mercury, xylene, water-ethanol mixture, etc., to replace the gaps of the materials or the gaps formed by material stacking, and indirectly calculate the porosity by measuring the true density of materials [5-6]. The liquid replacement method has the potential to absorb the replacement liquid for the granular materials with strong hygroscopic properties. At the same time, some particles have rough outer surface with hairs, irregular pores and folds. Under normal pressure, they cannot fully fill the entire pores due to the large surface tension of the liquid, resulting in large measurement deviation and narrow application [7-9]. Gas displacement method is one of the effective methods to measure porosity based on ideal gas state equation, and its measuring devices mainly include constant volume type [10-12], variable volume type [13] and comparative type [14]. The current measurement method
needs to change the measurement parameters several times to convert the actual volume of materials, and the measurement range with high accuracy is that the actual volume of materials accounts for 40%~70% of the sample container volume. It is necessary to change sample containers with different volumes when measuring different granular materials. The measurement accuracy and reliability of gas replacement method will be affected by sensor error, parts processing error, gas leakage and material filling rate, etc [15]. In order to eliminate the influence of variable operating condition parameters and material filling rate on the measurement results in the testing process, improve the measurement accuracy and reliability, and expand the measurement range, in this paper, based on the principle of ideal gas state equation test, a constant-volume measurement apparatus is designed to obtain the porosity of granular materials under the condition of constant pressure at one time, so as to realize the automatic control of the measurement process and automatic data collection. According to different measurement operating conditions, the measurement accuracy is verified by using different forms of granular materials. Under constant conditions, the measuring installation is calibrated by using high precision steel balls with dense surface, so as to eliminate the influence of filling rate on the measuring accuracy, and its measuring accuracy and reliability are verified by paddy experiment.

2. Principle and overall design of testing apparatus

2.1. Principle

The principle of porosity testing apparatus is shown in Figure 1.

![Figure 1. Schematic diagram of porosity testing apparatus.](image)

In the Figure 1, 1. Throttle speed regulating valve; 2. Check valve; 3. Container A; 4. Pressure gauge; 5. Throttle speed regulating valve; 6, 9, 11. Solenoid valve; 7. Container B; 8. Throttle speed regulating valve; 10. Air compressor.

Container A and container B are both rigid containers with same volume. When measuring, the container B is filled with the material under test, start the solenoid valve 9 to let the container B connected with the atmosphere. After the system opens the solenoid valve 11, solenoid valve 6 is automatically closed to control the air compressor to inflate the container A and the pressure data is transmitted to the processor in real time by the air pressure sensor. When it reaches the upper pressure limit, close the solenoid valve 11, wait for the air pressure of container A to stabilize, collect the gauge pressure and record it as p1, then the system automatically closes the solenoid valve 9, opens the solenoid valve 6, and wait for the air pressure of container A and container B to stabilize, then record it as p2, and obtain the porosity from the ideal gas state equation, which is:

\[
(p_1 + p)(V + V_1) = M_1RT
\]  

(1)
\[ p_a(V_k + V_2) = M_2RT \]  

(2)

\[ (p_2 + p_a)(V + V_k + V_1 + V_2) = MRT \]  

(3)

\[ M = M_1 + M_2 \]  

(4)

Combine the formulas of (1) ~ (4), we can obtain:

\[ \frac{(V_k + V_2)}{(V + V_1)} = \frac{(p_1 - p_2)}{p_1} \]  

(5)

Assume that \( \varepsilon = \frac{(p_1 - p_2)}{p_1} \), then there is:

\[ \frac{V_k}{V} = \varepsilon + \frac{\varepsilon V_1 - V_2}{V} \]  

(6)

- \( V \) — air volume in container A, cm\(^3\).
- \( V_k \) — air volume in container B, cm\(^3\).
- \( M_1 \) — mass of air in container A (including the air in the connecting pipe), g.
- \( M_2 \) — mass of air in container B (including the air in the connecting pipe), g.
- \( V_1 \) — volume of gas in connecting pipe of container A, cm\(^3\).
- \( V_2 \) — volume of gas in connecting pipe of container B, cm\(^3\).
- \( M \) — mass of air in container A and B (including the air in the connecting pipe), g.
- \( T \) — the aerothermodynamic temperature of air, K.
- \( R \) — air gas constant, 287.1 J/(kg×k).
- \( Pa \) — atmospheric pressure, Pa.
- \( \varepsilon \) — porosity, %.

In the formula (6), the volumes \( V_1 \) and \( V_2 \) are very small, which is less than one in ten thousand of the volume \( V \), and equation (6) can be simplified as:

\[ \frac{V_k}{V} = \varepsilon = \frac{p_1 - p_2}{p_2} \]  

(7)

2.2. Design of testing apparatus

The machining accuracy, airtightness of container A and container B, as well as errors caused by manual reading operation, will affect the measurement accuracy. This apparatus ensures the tightness of the apparatus and eliminates the error caused by human operation through the structural pressure-bearing seal design and electric control system.

![Figure 2. Construction diagram of pressure vessel.](image)

In the Figure 2, 1. End cap; 2. Upper flange; 3. Pneumatic connector; 4. Lower flange; 5. Pressure sensor; 6. O-ring seal.
2.2.1. Structural design of testing apparatus. In order to make the testing apparatus eliminate the introduction error of pipeline and have enough inflation upper limit pressure, the container volume design should be much larger than the pipeline volume and will not deform under the pressure of 1 MPa. The container structure is shown in Figure 2, and the design parameters are shown in Table 1.

Circumferential stress of cylinder:

\[
\sigma = \frac{pD}{2\delta} < \frac{[\sigma]}{n}
\]  

- \(\sigma\) — circumferential stress of cylinder, MPa.
- \([\sigma]\) — allowable stress of 304 stainless steel, \([\sigma]\) = 620 MPa.
- \(p\) — working pressure of pressure vessel, MPa.
- \(\delta\) — container wall thickness, mm.
- \(n\) — safety factor.
- \(D\) — outer diameter of vessel, mm.

| Parameter                        | Value                  |
|----------------------------------|------------------------|
| Material of container            | 304 stainless steel    |
| Outer diameter of container /mm  | 134                    |
| Inner diameter of container /mm  | 124                    |
| Height of cavity /mm             | 200                    |
| Thickness of container wall /mm  | 5                      |
| O-ring inner diameter × wire diameter /mm × mm | 140 × 3.5 |
| O-ring groove width /mm          | 4                      |
| O-ring groove depth /mm          | 2                      |
| O-ring compression ratio /%       | 28                     |

According to the design requirements, the maximum working pressure \(p = 1\) MPa, and the outer diameter \(D = 134\) mm, safety factor \(n = 8\), wall thickness \(\delta = 5\) mm, from the calculation we can obtain:

\[
\sigma = \frac{pD}{2\delta} = 13.4 < \frac{[\sigma]}{n} = 77.5
\]  

The circumferential stress is less than the allowable stress and meets the strength requirement.

The inner and outer wall surfaces of the container are smooth, and the upper and lower end surfaces are designed with fixing flanges. The lower flange is fixed on the bracket, and the upper flange is connected with the end cap. O-shaped sealing groove is designed on the end face of the upper flange, and the compression ratio of the sealing ring is 28%. The pneumatic joint is designed in the middle of the container.

2.2.2. Design of electric control system. The electronic control system is composed of upper computer, A/D analog-to-digital conversion module, pressure sensor, solenoid valve and power supply module, as shown in Figure 3.
Figure 3. Diagram of electric control system.

The PLC transmits the pressure sensor signal to the upper computer through the analog-to-digital conversion module, and the configuration software judges whether the container pressure reaches the set upper limit or balance value by inquiring the pressure value for many times. According to the set measuring program, the configuration software sends instructions to the PLC to control the switch of the solenoid valve and automatically control the measuring process. The interface developed by computer is shown in Figure 4.

Figure 4. Computer interface.

Table 2. Type and operating parameter of components

| Component                      | Model          | Operation range |
|--------------------------------|----------------|-----------------|
| Air compressor                 | OTS-550        | 0–0.7 MPa       |
| Pressure sensor                | SMC PSE560-02  | 0–1 MPa         |
| Two-position two-way solenoid valve | SMC AS2201F   | 0–0.6 MPa       |
| Throttle speed regulating valve | SMC KH06A-01S  | 0–1 MPa         |
| Filter pressure reducing valve | SMC AW20-02BG  | 0–1 MPa         |
| Pipeline                       | SMC outer diameter 4mm | 0–1 MPa |
| PLC                            | Panasonic FP0  | 24 V            |
| A/D conversion module          | Panasonic AFP0480 | 0–5 V         |

The interface is divided into apparatus dynamic feedback, parameter setting and display, and data saving. The machine can feedback the measured value of state parameters of the apparatus in dynamic loading and unloading process and equilibrium state in real time, and output the open and closed state and airflow flow situation of each solenoid valve. The input quantity of the system is the upper limit
pressure of container inflation, the control quantity is inflation pressure, the detection quantity is pressure, and the output quantity is real-time pressure, opening and closing state and porosity of each valve of the apparatus. The controller of the control system itself is a complete independent control unit and a basic data acquisition node, which realizes automatic data storage. When measuring, click the start button on the man-machine interface, and the apparatus will automatically run until the porosity is obtained. Specific design parameters and main components are shown in Table 2.

3. Test and result analysis

3.1. Orthogonal test

3.1.1. Test of measuring apparatus and analysis of results. Standard stainless-steel spheres, plastic quadrilateral prisms, cobblestones and small stones with different shapes can be used as test materials. Dry cobblestones and small stones in a drying oven to remove volatiles. The volume of each test sample is measured by calculation and drainage method, and the porosity of the test sample is set according to the volume of pressure vessel, as shown in Table 3. The physical object of the test measuring device is shown in Figure 5.

Table 3. Porosity of test samples

| materials         | Size /mm | Porosity level |
|-------------------|----------|----------------|
|                   |          | 1     | 2    | 3     | 4     |
| Stainless steel sphere | 30       | 50.20 | 64.86 | 80.09 | 94.73 |
| Quadrilateral prism | 29 ×23 ×37 | 51.94 | 65.52 | 80.10 | 94.78 |
| Cobblestone       | 25 ～40  | 50.99 | 66.08 | 80.48 | 95.05 |
| Small stone       | 5 ～10   | 50.34 | 66.87 | 81.45 | 95.02 |

Figure 5. Porosity testing apparatus.

3.1.2. Test method. In addition to sensor error and system error, the factors affecting the measurement accuracy of porosity testing apparatus, such as material configuration to be measured, inflation pressure, throttle and deceleration valve opening between containers, may bring errors to the measurement system as well. Different porosity and inflation pressure will affect the final equilibrium pressure and produce systematic errors. Different throttle valve opening will change the airflow speed and inflation time in the inflation process, resulting in pressure drop between containers. Different material states lead to the change of material surface characteristics in the measurement process, and dense, smooth or rough porosity may cause differences in the aeration process. In order to verify the
influence of these factors, the measurement error $\Delta$ and measurement repeatability $S^2$ are used to 
measure the main evaluation indexes of the testing apparatus performance.

Measurement error:

$$\Delta = x - L$$  \hspace{1cm} (10)$$

- $x$ — measured value, %.
- $L$ — theoretical calculated value, %.

Measurement repeatability:

A set of data can be obtained by repeated measurement under the same experimental conditions. The repeatability of variance reflecting the dispersion degree of the data. The greater the variance, the worse the repeatability. The repeatability variance is:

$$S^2 = \frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1}$$  \hspace{1cm} (11)$$

- $x_i$ — measured value.
- $\bar{x}$ — Mean.

In order to find out the primary and secondary factors affecting the measurement performance, according to the evaluation index of the porosity testing apparatus, the porosity of the material to be measured, the shape of the object to be measured, the opening of the speed regulating throttle valve of the communicating container and the inflation pressure are selected for the orthogonal test of four factors and four levels. Test factors and levels are shown in Table 4.

### Table 4. Porosity of test samples

| Level | Factors | Porosity /% | Material         | Throttle valve opening degree % | Inflation pressure /MPa |
|-------|---------|-------------|------------------|---------------------------------|------------------------|
| 1     | 50      | Stainless steel sphere | 25              | 0.1                             |
| 2     | 65      | Quadrilateral prism    | 50              | 0.2                             |
| 3     | 80      | Cobblestone           | 75              | 0.3                             |
| 4     | 95      | Small stone           | 100             | 0.4                             |

3.1.3. Test results. Choose orthogonal test design table $L_{16}(4^5)$, and leave an empty column as an estimate calculate the test error, the measurement error is the mean error after repeated 10 times under the same test conditions, and the repeatability is the variance of this group of data. The results of orthogonal test are shown in Table 5.

### Table 5. Results of orthogonal experiment

| Test serial number | Porosity /% | Material        | Throttle valve Opening Degree /% | Inflation Pressure /MPa | empty columns | Test index | Measurement error | Repetitive error |
|--------------------|-------------|-----------------|----------------------------------|-------------------------|---------------|------------|-------------------|------------------|
| 1                  | 50          | Sphere          | 25                               | 0.1                     | 1             | 1.1271     | 0.2403            |
| 2                  | 80          | Cobblestone     | 25                               | 0.3                     | 3             | 0.1879     | 0.0598            |
| 3                  | 95          | Small stone     | 25                               | 0.4                     | 4             | -0.4236    | 0.0018            |
| 4                  | 65          | Quadrangular prism | 25                            | 0.2                     | 2             | 0.9196     | 0.0862            |
| 5                  | 65          | Small stone     | 75                               | 0.1                     | 3             | 1.3517     | 0.1363            |
| 6                  | 95          | Cobblestone     | 50                               | 0.1                     | 2             | -0.3246    | 0.2844            |
| 7                  | 80          | Quadrangular prism | 100                           | 0.1                     | 4             | 0.6232     | 0.5148            |
| 8                  | 50          | Small stone     | 100                              | 0.3                     | 2             | 1.1596     | 0.0328            |
3.1.4. Analysis of variance of test results. The results of variance analysis of Table 5 are shown in Table 6.

It can be seen from Table 6 that the main factors affecting the measurement error and repeatability of the testing apparatus are porosity and inflation pressure of the material to be measured. Assuming that the error between container A and container B is $e_v$, there is:

$$V_A = V_B + e_v$$

(12)

According to formula (12), $V$ involved in calculation is the volume of container A, and the actual porosity is:

$$\varepsilon_{ture} = \frac{V_k}{V_B}$$

(13)

| Measurement error | Measuring repeatability |
|-------------------|------------------------|
| Porosity | Material | Throttle valve opening degree | Inflation pressure | Porosity | Material | Throttle valve opening degree | Inflation pressure |
| Freedom degree | 3 | 3 | 3 | 3 | 3 | 3 | 3 |
| The F value | 81.813 | 5.493 | 0.702 | 3.038 | 1.817 | 1.911 | 1.48 | 46.205 |
| The Sig. value significant | 0.002 | 0.098 | 0.611 | 0.193 | 0.318 | 0.304 | 0.378 | 0.025 |

Measurement error:

$$\Delta = \varepsilon - \varepsilon_{ture} = \frac{V_k}{V_A} - \frac{V_k}{V_B} + e$$

(14)

It can be obtained from formulas (8), (12) ~ (14) that:

$$\Delta = \varepsilon \left(1 - \frac{V_A + e_v}{V_B}\right) + e$$

(15)

- $V_A$ — Volume of container A.
- $V_B$ — Volume of container B.
- $\varepsilon$ — Sum of other errors.
- $e_v$ — Container processing error.

According to formula (15), the system error of the testing apparatus follows a certain rule and can be effectively eliminated by compensation. As can be seen from Table 6, different material configuration, throttle valve opening and inflation pressure have no significant influence on the measurement error of the testing apparatus, and the testing apparatus can meet the requirements of measuring the porosity of different kinds of materials.
The results of estimating inflation pressure factor and measuring marginal mean of repeatability index from test results are shown in Table 7.

| Inflation pressure/MPa | Repeatability |
|------------------------|---------------|
| 0.4                    | 0.017         |
| 0.3                    | 0.040         |
| 0.2                    | 0.067         |
| 0.1                    | 0.294         |

It can be seen from Table 7 that the higher the inflation pressure, the smaller the variance of repeated test data and the better the repeatability. After introducing the sensor error, it can be obtained by formula (7):

$$
\varepsilon = \frac{p_1 - p_2 + e_{p_1} - e_{p_2}}{p_2 + e_{p_2}} = \frac{p_1 - p_2}{p_2 + e_{p_2}} + \frac{e_{p_1} - e_{p_2}}{p_2 + e_{p_2}}
$$  \hspace{1cm} (16)

- $e_{p_1}$ — measurement error of inflation pressure.
- $e_{p_2}$ — measurement error of balance pressure.

It can be known from equation (16) that the higher the inflation pressure $p_1$ is, the higher the pressure $p_2$ during equilibrium, and the smaller the influence of error on the calculation result, the more stable the measurement is. Since the sensor error changes randomly, it can't be eliminated, but the random error can be reduced by choosing appropriate inflation pressure. In view of the normal working pressure range of each component, the optimal inflation pressure of the testing apparatus is 0.4 MPa.

### 3.2. Calibration test of testing apparatus

#### 3.2.1. Test materials and methods

The calibration materials are $\varnothing = (30 \pm 0.02)$ mm high precision stainless steel balls with high surface compactness and plastic cylinders with a certain volume. By accurately measuring the volume of container B of the measuring device, the stainless-steel ball and plastic cylinder are used to produce the test samples with porosity of 18.75%, 26.80%, 41.43%, 50.20%, 60.17%, 70.13%, 80.32% and 90.04%, respectively. Set the upper inflation pressure of the testing apparatus to 0.4 MPa, and measure samples with various porosity values for 15 times respectively, and compare the obtained data with the theoretical values, so as to obtain the error variation law of the apparatus and compensate it.

#### 3.2.2. Test results and analysis

The calibration test results of the testing apparatus are shown in Table 8.

| Theoretical porosity /% | Measurement average porosity /% | Measurement error /% |
|-------------------------|---------------------------------|----------------------|
| 18.75                   | 20.19                           | 1.44                 |
| 26.8                    | 28.18                           | 1.38                 |
| 41.43                   | 42.29                           | 0.86                 |
| 50.2                    | 50.9                            | 0.7                  |
| 60.17                   | 60.71                           | 0.54                 |
| 70.13                   | 70.52                           | 0.39                 |
| 80.32                   | 80.6                            | 0.28                 |
| 90.04                   | 89.72                           | -0.32                |
| 100                     | 99.05                           | -0.95                |
It can be seen from Table 8 that the measurement error decreases monotonously with the increase of porosity, which accords with the analysis result of Formula (15) and can be determined as the systematic error of the testing apparatus and eliminated by error compensation. The results of fitting and analysing the test values by using Matlab data processing software are shown in Figure 6.

The compensation formula for the variation of the obtained error with the measured porosity is:

$$\Delta \varepsilon = -0.00001078x^3 + 0.001723x^2 - 0.105x + 3.046$$

(17)

- $x$ — measuring porosity.
- $\Delta \varepsilon$ — deviation value.

The measurement results are modified based on the compensation formula and configuration software. The test samples with the theoretical porosity of 16.02%, 25.14%, 35.57%, 49.05%, 58.42%, 67.79%, 76.57%, 85.36% and 94.14% were used for matching the stainless-steel ball and stainless-steel cylinder. The average value of each sample was measured five times with the compensated device, and the measurement error after and before compensation was shown in Figure 7.

It can be seen that the maximum variation range of error after compensation is less than 0.3%, which proves the effectiveness of the compensation scheme.

4. Conclusion
The text of your paper should be formatted as follows:

(1) On the basis of the ideal gas state equation, an apparatus for measuring the porosity of constant volume granular materials is designed, which realizes the automation of the measurement process real-time display and storage of control and detection parameters, simple structure, convenient operation and high measurement reliability.

(2) The variation law of measurement error with porosity value is obtained by experiment, and the
calculation formula of compensation coefficient is offered. Under the condition that the upper limit of inflation pressure is 0.4 MPa, stainless steel balls and cylinders are used, and the filling rates are 16.02%, 25.14%, 35.57%, 49.05%, 58.42%, 67.79%, 76.57%, 85.36%, 94.14%. The test results show that the range of measurement error is less than 0.3%.

(3) The inflation pressure has a certain influence on the measurement accuracy. In the set measurement range of 0.1~0.4 MPa, the higher the inflation pressure, the smaller the measurement error and the better the repeatability.

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