A MANOMETRIC METHOD OF MEASURING THE
APPARENT DENSITY OF GRANULAR MATERIALS

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Results from theoretical and experimental studies of the manometric method of monitoring the density of granular chemisorbents in a complex determination of their thermophysical characteristics are presented. The processes that occur in the course of measurements are described, the static characteristic of the measuring plant is investigated, the errors are determined, and computational dependences and recommendations on the use of the method are presented. The mutual influence of the processes involved in the measurement of thermal conductivity and density is estimated. It is established that the apparent density of granular materials may be monitored by means of a relative manometric method with compensation for the influence of atmospheric pressure on the measurement results.

Keywords: *granular material, porous material, volumetric density.*

Materials in the form of heterogeneous gas–solid systems are widely used in modern industrial production and agriculture. In order to assure a high level of performance of technological processes when such materials are used, it is necessary to know their thermophysical properties, such as density, thermal conductivity, thermal capacity, and thermal diffusivity [1–3].

The density of granular material is determined by measuring its mass and volume. Different methods and devices are used to measure mass [4]. The mass of the hardest particles or the mass of the particles together with the air between them may be measured. Because of the great difference between the density of a gas and that of particles, the error of such measurements will be insignificant. The problem of measuring the volume of granular material does not have a unique solution. Depending on the relationship between the solid and gas phase in a volume that is being monitored, we distinguish the packed, volume, apparent, and effective density of granular material [1, 2]. By the volume density is understood the average density of the particles of a material in the volume of which there are closed and open pores. The definition of the apparent density in a volume of particles does not include open pores. The effective density is the density of the matter of the solid particles. For porous materials, some of these types of density may coincide, depending on the structure of these materials.

The volume density and apparent density are measured by the methods of liquid and gas substitution. Methods of liquid substitution suffer from a number of drawbacks as compared to gas substitution, for example, the need to remove gas bubbles through heating or other means; interaction of the liquid with solid matter, leading to a variation in the solid volume and the fact that the liquid cannot be reused; the need to take into account the molecular volume of the liquid to assure it can percolate through open pores [2]; and the need to dry the material and the measurement tank.

The methods used to measure density with gas substitution are based on the variation of the pressure in the measurement tank containing the test substance. Nondestructive methods of gas substitution are sometimes classified as noncontact methods, since in the course of compressing the gas, which is a constituent part of granular material, no other medium comes into contact with the solid particles.

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The critical nature of experimental investigations of pneumatic methods of measuring density has grown as a result of their use in complex measurement of the therrmophysical characteristics of granular materials [3, 5]. Traditionally, the effective thermophysical characteristics of the poured layer are determined when monitoring granular materials [6]. However, in actual practice it is often necessary to separately determine the heat transport that arises as a result of convection and thermal conductivity. Measurement of the apparent density of the test material is one of the important steps in the solution of this problem. The practical use of the method of gas substitution for this purpose has led to the need to investigate the factors that affect the measurement precision. In the present study, we will present the results of these investigations.

The methods of gas substitution include pneumodynamic methods [7–9] and manometric methods [9–13]. The latter do not require connections to a source of compressed gas and are distinguished by a fewer number of parameters that affect the result of the measurements.

In manometric methods, the volume of a substance is estimated from the variation in the pressure in a pneumatic system as its volume varies to a given value [1, 2]. A plant that implements the manometric method comprises a measurement tank for the test material connected to a tank of variable volume and a manometer [3, 5, 9–12].

Let us consider two modifications of the manometric method of measuring the apparent density. In the first modification, a tank of variable volume is compressed to a given magnitude and a judgement as to the volume of granular material is arrived at from the variation in the pressure [1–3, 5, 11]. The second modification presupposes a determination of the volume of the material from the variation in the volume of the tank necessary for attaining a given pressure [12, 14]. In both modifications, the readings of the devices must be established after a steady state in which heat is released into the environment has been attained [13]. In this case, there is no need to perform an analysis of heat liberation or complex computations as in [12], where the readings were determined immediately after the sensor (pressure indicator) had been actuated.

If no special measures are adopted, the process of compressing a gas in a pneumatic system will be polytropic, i.e., the laws of isothermal and adiabatic processes will not be applicable [12]. In a polytropic process of compression of a gas, its temperature and pressure grow. Following completion of the compression, the gas releases heat to the walls of the vessel and the test substance. As a result, over the course of time the temperatures of the walls and the test substance are equalized and the pressure decreases. Graphs illustrating the variation in the pressure in the measurement tank following compression of a gas are presented in [3], and from an analysis of the graphs it follows that maximum filling of the volume of the tank should be achieved to simplify the measurement process. The most of the volume of compressed gas continuously releases heat to the particles of the material and the compression process approaches an isothermal process.

Temperature is the most highly informative indicator in methods used to monitor the thermophysical characteristics of substances [3, 5, 6]. In the course of compression, a gas warms up and the corresponding parameters of the thermal process vary, becoming a source of errors in the determination of density. However, in the course of repeated measurements the heating – cooling processes occur cyclically as a consequence of variable compression – distension of the tank and, on average, heat is not released.

Following cooling of the gas to the ambient temperature, its pressure will correspond to the pressure following compression in an isothermal process and, consequently, it is permissible to apply the Boyle–Mariotte law in the computations. Cooling of a gas takes a certain length of time, hence the use of the manometric method requires maintaining the pneumatic system hermetically sealed. Leakages lead to a decrease in the pressure following compression of the gas, introducing thereby a substantial contribution to the measurement error. Even micro-leakages make it difficult to establish a steady-state regime.

Continuous heating of a granular material is required in complex determination of the thermophysical characteristics of such material [3, 5]. Under these conditions, it is impossible to assure isothermal compression of a gas for the purpose of measuring the density. From the results of experiments, the pressure in a pneumatic system 120 ml in volume is established in a period of time not exceeding 10 sec. At a rate of heating of 0.1 K/sec the variation in the temperature over this period of time amounts to 1 K. Using an MRX5050GP maonometer [3] with upper measurement limit 50 kPa, a gauge pressure in the pneumatic system of approximately 40 kPa should be selected. Following compression of the gas, the absolute pressure in a measurement of the apparent pressure will be around 140 kPa. Heating by 1 K in an isochoric process leads to a variation in the pressure by roughly 470 Pa, which amounts to 1.2% of the variation in the pressure (40 kPa) as a result of a variation in the volume of the pneumatic system. In the case of a high rate of heating, the error will grow proportionally. Corrections may be
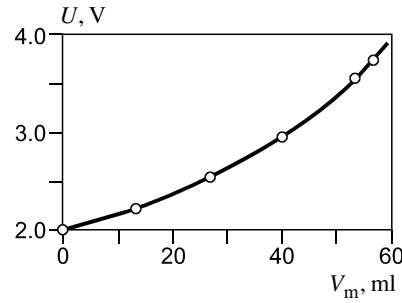
Fig. 1. Static characteristic of plant for measurement of the
apparent density of granular materials.

introduced or the gas compressed to higher pressures as a way of reducing the influence of heating of a material on the result of a measurement of the apparent density. The latter alternative should not be used due to complication of the hermetic sealing of the measurement tank and low efficiency.

In accordance with the Boyle–Mariotte law, we may write the following equation:

$$p_0(V_0 - V_m + \Delta V) = (p_0 + \Delta p)(V_0 - V_m + V_d), \tag{1}$$

where p_0 is atmospheric pressure; V_0, volume of pneumatic system following compression of a variable-volume tank consisting of the volume of the measurement tank, the connecting pipes, and the cavity of the manometer; V_m, volume of granular material; ΔV, variation in volume upon compression of the gas; Δp, variation in pressure in the pneumatic system upon compression; and ΔV_d, variation in volume as a consequence of deformation of the elements of the pneumatic system upon compression of gas. We will set $\Delta V_d = k_d \Delta V$, where k_d is a proportionality factor. From (1), we obtain:

$$\Delta p = p_0(\Delta V - k_d \Delta V)/(V_0 - V_m + k_d \Delta V). \tag{2}$$

From Eq. (2), it follows that Δp is directly proportional to p_0 and ΔV with $k_d = 0$, and that the dependence of Δp on the volume of granular material is nonlinear. From the proportionality of the two quantities Δp and ΔV, it follows that there are no procedural assumptions that have to be made for the selection of the values of these quantities in an actual measurement plant. To decrease leakages, values of Δp and ΔV should be selected as the least possible on the basis of the range of measurements of the manometer employed. However, because of heating of the granular material, in comprehensive monitoring of the thermophysical properties the minimum value of Δp should be selected so that it is at least not less than the variation in the pressure caused by heating of the material. Otherwise, it will be difficult to assure effective compensation of the latter.

With $\Delta V = 0.4(V_0 - V_m)$ and $k_d = 0.1$, the maximum deviation of the dependence $\Delta p(\Delta V)$ from the proportional will not exceed 2% of the average value of Δp. Therefore, without significant loss of precision, Eq. (2) may be written in the form:

$$\Delta p = p_0 \Delta V/(V_0 - V_m), \tag{3}$$

where the volume V_0 is an empirical constant. Experimental determination of V_0 is necessary, since it is complicated process to determine the effective volume of a pneumatic system with high degree of precision using any other method.

From the results of experiments with an empty tank, it was established that the maximum deviation of the experimental dependence $\Delta p(\Delta V)$ from the proportional does not exceed 1.6% of the average value of the pressure. Random deviations represent the basic component. In experiments the variation of the volume ΔV was in the range 10–160 ml, while the variation in the pressure Δp in the range 4–60 kPa.

Figure 1 represents the dependence of the output volume of an MPX5050GP manometer with electrical output signal on the volume of granular material in the measurement tank, which is a static characteristic of the measurement plant.

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TABLE 1. Results of Experimental Determination of the Error in the Measurement of the Apparent Density of Granular Materials

Material	Average diameter of granules, mm	Density, kg/m³		Error	
		effective	measured	absolute, kg/m³	relative, %
Glass	1.7	2610	2617	7	0.3
Caprolan	2.0	1146	1164	18	1.6
Polyvinylchloride	4.0	1243	1234	9	0.7
Porcelain	5.5	2295	2421	126	5.5
Porous ceramic	6.5	1983	4849	2866	146

The manometer's output signal is related to the gauge pressure by a linear function, hence in calibration and operation of the measurement plant it is sufficient to carry out all the calculations with the use of measured values of the voltage.

Experimental data were obtained by filling the measurement tank with lead shot 6 mm in diameter. The volume of the shot was determined from the mass of the suspension and the density, which was measured by the method of liquid substitution. The points on the static characteristic (Fig. 1) represent the arithmetic mean of the result of observations while the line represents the results of an approximation of the experimental data by the function

$$U = U_0 \Delta V / (V_0 - V_m), \qquad (4)$$

where U and U_0 are the voltages at the manometer output corresponding to the pressures Δp and p_0.

Approximation is performed by the method of least squares using the linear function $y = b_0 + b_1 V_m$ with the substitution of variables $y = U^{-1}$, $b_0 = V_0/(U_0 \Delta V)$, $b_1 = -(U_0 \Delta V)^{-1}$. Following calculation of the coefficients b_0 and b_1, the values $U_0 \Delta V = -b_1^{-1} = 0.2387$ V·m³ and $V_0 = U_0 \Delta V b_0 = 0.1201$ m³ are determined on the basis of experimental data for y and V_m. The maximum absolute deviation of the measured value of the voltage U_i, where i is the ordinal number of the measurement, from the theoretical $U(U_{mi})$ (4) does not exceed 5 mV, the quadratic deviation does not exceed 3.4 mV, and the relative error of the approximation does not exceed 0.12%.

The (4) function is nonlinear, and from the graph (cf. Fig. 1) it follows that the greatest sensitivity of the plant to variations in the volume of granular material V_m is typical of its maximum values. Therefore, when carrying out measurements the measurement tank should be filled to the maximum extent possible with the test granular material.

After obtaining the experimental coefficients of the static characteristic (4) of a plant for the manometric method of measuring the apparent density of granular materials with identical atmospheric pressure for different materials, the error of the measurement was determined (Table 1). The effective values of the density were obtained by the method of liquid substitution. Water was used as the immersion liquid. A number of measures were adopted to prevent the formation of gas bubbles and to remove any gas bubbles that did form.

From the table, it follows that for materials that are not characterized by highly developed porosity (glass, plastic), the error in the measurements is comparatively low. In most cases, the values of the density determined by the method of gas substitution are higher than those obtained by the method of liquid substitution. Similar differences are observed in the data presented in [12]. Results of measurements of the density of ceramic articles possessing internal pores that often communicate with the atmosphere are presented in the table. The movement of liquid through such pores is hampered by surface forces and the forces of viscous friction, the magnitudes of which are much less in the case of gas penetration. For this reason, the results of measurements of the density of granular materials by the method of liquid and gas substitution differ substantially. If the method of liquid substitution is used for such materials, an appropriate liquid must be selected.

By Eq. (3), the error in measurements of the volume of particles of granular material by the manometric method associated with variations in atmospheric pressure in the range 96–104 kPa reaches ±4%. Differential [12–14] and relative [1, 2, 10, 11] methods of measurement are used to reduce the influence of atmospheric pressure on the result. Differential methods in which
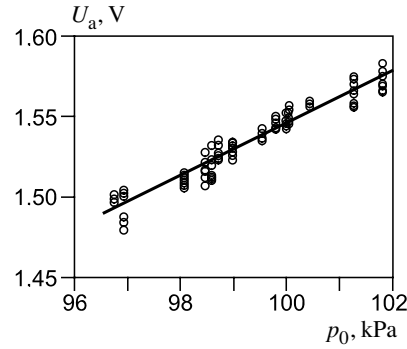
Fig. 2. Dependence of output voltage U_a of manometer
on atmospheric pressure p_0.

air is compressed in the measurement and comparison chambers and the pressure drop in the chambers measured differ in terms of the complexity of the technical implementation and in the computational dependences. In the relative methods, the atmospheric pressure is determined prior to determining the volume of particles of the granular material by compressing the gas in the empty measurement tank.

For an empty measurement tank ($V_m = 0$), we obtain from (4):

$$U_a(U_0) = U_0 \Delta V / V_0, \tag{5}$$

where U_a is the voltage, which depends on atmospheric pressure.

The dependence of the voltage at the output of an MPX5050GP manometer on atmospheric pressure obtained from the results of observations over several months is presented in Fig. 2. The experimental dependence is approximated by the function $U_r = -0.083 + 0.016 p_0$, where voltage is expressed in terms of volts and pressure in terms of kilopascals. The modulus of the absolute deviation of the function from the experimental data amounts to 16 mV, the root-mean-square error is 6.1 mV, and the relative error of the approximation is 0.4%.

The graph represented in Fig. 2 demonstrates the value of using the relative manometric method to measure the density of granular materials to compensate for the influence of atmospheric pressure on the result, since a technical-grade manometer used in the actual physical process may be employed to establish the natural oscillations of the affecting quantity [3]. During the period in which the experiments are conducted, besides atmospheric pressure the temperature and air humidity are also measured. In accordance with a theoretical analysis, these parameters do not have any effect on the result of measurements. The graph in Fig. 2 confirms this, since there is no substantial scatter in the experimental data with the same atmospheric pressure.

From Eq. (4), we may derive a formula for determining the volume of particles of a granular material:

$$V_m = (UV_0 - U_0 \Delta V)/U, \tag{6}$$

where the voltage U_0 may be calculated on the basis of the value of the voltage U_a obtained for an empty tank. Substituting U_0 from formula (5) into formula (6) we find

$$V_m = V_0(1 - U_a/U), \tag{7}$$

where the volume V_0 is determined in an experiment with a body of known volume. A formula analogous to (7) is presented in [2] for the case of compression of a gas by the effect of the pressure of a column of liquid.

With the use of the relative method with compensation for atmospheric pressure, a measurement error less than 1% can be achieved. The relative method also makes it possible to simplify the calculations and quickly vary the value of ΔV as a function of the porosity of the test material without any calibration.

The method of measuring the apparent density of granular and porous materials that has been considered here may be successfully used to determine the densities of solid bodies of complex form and heterogeneous systems in any branch of industry.

The following conclusions may be arrived at from the results of the study:

1) it is necessary to establish the readings of the manometer in the steady state after the gas has released heat produced as a result of compression;

2) the relative manometric method, which assures compensation for the effect of atmospheric pressure on the measurement result and simplifies the calculations, should be used in measuring the apparent density;

3) the experimental data are approximated by theoretical dependences with relative error not greater than 0.4%, which attests to the high degree of precision of the manometric method;

4) in the manometric method, the precision is often limited by the feasibility of hermetic sealing of the pneumatic system in an actual measurement plant; in order to achieve a measurement error below 1%, considerable attention should be devoted to questions of air tightness and constancy of the volume of the pneumatic system;

5) heating of the gas in the course of compression and heating of granular material in the course of measurements of thermophysical properties exert a mutual influence, which requires the introduction of corrections into the measurement results; and

6) in order to reduce leakages, air may be compressed into a minimum volume determined by the range of measurement of the manometer employed, though to achieve effective correction of the results of measurements of the apparent density, the variation in the pressure due to compression must not be less than the variation of pressure caused by heating of granular material in the course of a measurement of its thermophysical properties.

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