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MODIFIED WATER DISPLACEMENT METHOD AND ITS USE FOR DETERMINATION OF BULK DENSITY OF POROUS MATERIALS

(METODE MODIFIKASI PERPINDAHAN AIR DAN PENGGUNAANNYA UNTUK MENENTUKAN MASSA JENIS MATERIAL BERPORI)

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ABSTRACT

In this research work, a modified water displacement method (MWDM) was designed and used in addition to geometry method (GM) to measure the bulk volume and then determine the bulk density values of asbestos ceiling board, cardboard paper, chalk, clay (compacted) and gypsum board that have been sun-dried to constant weight. The mean bulk densities determined by both methods were compared with the reference bulk density values of the same porous materials obtained in this work using standard test procedure in accordance with ASTM D6683-14. It was observed that, for all the tested porous materials, the percentage error in the mean bulk density values ranged from 2.3% to 49.6% when using GM and 0.9% to 5.7% by using the MWDM. Also, at 0.05 level of significance with a degree of freedom of 3, correlation coefficients of 0.7430 and 0.9955 were obtained in the cases of GM and the MWDM respectively. Again, all other analyses performed similarly revealed that the mean bulk densities obtained by the MWDM only were in close agreement with their corresponding reference values, thereby implying that apart from being cost-effective, the MWDM is better than GM in terms of accuracy, reliability, and validity. More importantly, it is noteworthy that even if the glass cylinder available for use is ungraduated, this MWDM can be employed to obtain accurate, reliable and valid bulk density values of porous materials in order to enhance thorough physical characterization, proper selection and suitable applications of such materials.

Keywords: Bulk Density, Internal Diameter, Material Characterization, Porous Materials, Water Displacement, and Water Proofing

INTRODUCTION

The word material simply refers to a substance, composed of matter that is intended for use in certain applications in order to achieve the desired result. Since matter can exist as solid, it implies that a material can be in solid form and so, based on the properties of the solid state of matter, a solid may be defined as matter sample that is capable of retaining its own shape and density when not confined. Solids that have minute interstices through which air or liquid may pass easily are referred to as porous materials. Apart from the three main groups of porous material namely, porous metals, porous ceramics and polymer foams (Gue-Feng & Peisheng 2014), porous materials are also classified as microporous, mesoporous and macroporous materials on the basis of the size of their pore diameters. In accordance with the standard specified by the International Union of Pure and Applied Chemistry (IUPAC), porous materials with pore diameters less than 2nm are regarded as microporous materials, those whose pore diameters are between 2nm and 50nm in size are classified as mesoporous materials, while macroporous materials are considered to be those of pore diameters greater than 50nm in size. As reported by Zdravkov et al 2007, in their research work, classification of pores is one of the basic requisites of comprehensive characterization of porous solids. Generally, porous materials have properties that vary depending on many factors among which are composition, length scales, impurities, surface structure, arrangement and shape of the pores, etc.

In the 19th Century, a major breakthrough occurred in the understanding of materials through the demonstration performed by an American Scientist named Josiah Willard Gibbs. Therefore, on the strength of such consideration, it is very obvious that the ability to understand the existing relationship between the aforementioned factors and the properties of a material has been playing very vital rule in most of mankind's technological breakthroughs, since then till date. Observably, what was considered a defect on solid materials some years ago are today being utilized advantageously based on the properties obtained from microporous and mesoporous materials. For instance, since the manufacturing of a material of perfect crystal is physically impossible, material scientists have been using various scientific techniques to manipulate defects such as precipitates, grain boundaries, interstitial atoms, vacancies or substitution atoms in crystalline materials to produce porous materials of desired properties. One of such techniques is the micro-molding method adopted by Soumaya et al 2015 in fabricating porous polymethyl hydrosiloxane structure using porous silicon scaffolds. Several other researchers including but not limited to Aizawa 2018, Udeni et al 2016, Xiao-Yu 2017, He et al 2012, Jang et al 2014 and Jun et al 2017, have, in their research reports, also confirmed the possibility of designing and developing useful porous materials of desired properties.

In everyday life, the emerging properties of enormous practical importance exhibited by fabricated porous materials have led to a pronounced increase in the range of their applications from applied sciences to medical diagnosis and engineering. For example, while Guo et al 2016 observed that high density, high mechanical strength, and thermal stability have made activated charcoal the best choice for adsorption, it has been found and reported by Sun et al2016, that the significant effort devoted to the applications, from energy storage and conversion, catalysis, photocatalysis, adsorption, separation, and sensing to biomedicine, of hierarchically structured porous materials has been as a result of their high surface area, excellent accessibility to active sites, and enhanced mass transport and diffusion. In their research work, Wang et al 2017 have posited that due to the possession of special physiochemical properties, synthesized inorganic porous materials are useful in environmental protection for removal of mobile source pollution. It is also worthy to mention that porous materials can be used in the reduction of aerodynamic sound (Takeshi et al 2010), removal of simazine from water (Esposito et al 2016) and water processing (Xu et al 2018). Though the size control, density, channel conductivity, roughness and surface functionalization of microporous, mesoporous and macroporous materials are keys for their future applications, the use of such materials has become a practical approach in reaching several goals in medical applications like drug delivery, surgical implants, medical imaging, biological sensors, etc. Such practical possibilities are due to their performance under ambient conditions as well as high mechanical and thermal strength under harsh conditions (Michailidis et al 2014; Solano-Umana & Vega-Baudrit 2015; Chatap & Patil 2017; Lett et al 2018). According to Gunathilake et al 2017, the advantages of developed biomaterials have outweighed the limitations of synthetic materials for applications in fields like biomedical and microbiology day by day.

Moreso, tremendous progress has been made in the energy sector in the past decades through the application of fabricated porous materials to improve the energy efficiency of buildings (Rashidi 2018), provide a source for renewable energy (Roshandel et al 2015), store and convert energy (Vekariya et al 2018; Li et al 2012), construct fuel cell electrodes (Brandon & Brett 2006; Wejrzanowski et al 2016), etc. Such great advancement has also been observed in the area of thermal management where developed porous materials are used as thermal barriers or heat exchangers (Clyne et al 2006), good heat insulating materials (Du et al 2008; Bai et al 2017), heat absorbers (Nakajima 2010), and can as well be applied to maintain a uniform temperature distribution, increase heat transfer rate, control reaction rates and improve heat flux absorption in thermal and chemical systems depending on their ultimate design (Torabi et al 2017). In all cases of applications, the selection of suitable porous materials is only possible through proper characterization, without which no scientific understanding of their properties could be ascertained. Also, in probing the properties of such materials for comprehensive characterization, density remains a very vital physical parameter to be determined as far as it continues to play a major role in the development of new materials, the influence of mechanical properties and help to address the functionality of the materials.

To determine the density of a material of any shape, both the mass and volume of the material must be known. While measurement of a material’s mass can be done easily with the use of a weighing balance, that of the volume is not so since the method to be employed depends on the shape of the material in question. In the laboratory, a method of geometry and water displacement method are commonly and often used to obtain the volume and then compute the density of non-porous materials such that the results obtained compare very well in accuracy, validity, and reliability with those determined using costly, uncommon and advanced techniques. However, for porous materials, the use of geometry method may yield results but with limited accuracy. Also, the use of the traditional water displacement method as described by (Kessler et al 2010; Anyakoha 2013; Ikeobi et al 2011), is obviously, unsuitable since it will enable the porous material to absorb the water as they remain in contact with each other even for few seconds. Therefore, even though water is a common laboratory liquid and as such, its use in measuring the bulk volume of a porous material for bulk density determination would be cost-effective, the above mentioned drawback points to the fact that there still remains a very serious challenge capable of preventing the use of such common and cost-effective technique to perform bulk density determination for physical characterization of porous materials thereby limiting their applications especially when the change of structures is involved.

Hence, this research work has been designed to address how to overcome the observed challenge by using a modified form of the traditional water displacement method to measure the bulk volume and then determine the bulk density of porous materials. Since a materials' property may be a function of one or more independent variables, this study, specifically, aims at determining the bulk density values of some porous materials using geometry technique and the modified water displacement method, and then comparing the results with the ones obtained as reference values using a standard test method in order to establish the data quality and effectiveness of the new technique.

THEORY

Density

This is the physical property of a material that is used to express the quantity of matter the material has to its volume. Depending on the degree of compactness of matter, density varies from one material to another. True, or apparent or even bulk density is often calculated as the ratio of mass to volume of a material, and it can be expressed mathematically as

$$\rho = \frac{m}{V} \quad (1)$$

where

m = mass of the material

V = true or apparent or bulk volume of the material

ρ = true, apparent or bulk density of the material

Apart from illustrating how density relates with mass and volume of a material, the equation above clearly shows that before the density of a material can be determined, the mass and volume of the material must be known.

Geometry Method

This technique is used to determine the volume of a regularly shaped material. It involves measurement of the dimensions needed for calculation of a material's volume using a suitable mathematical formula. Since materials can be cut to have different regular shapes, one mathematical formula cannot be used to find the volume in all the cases. For example, the volume of a material that is in the form of a cuboid can be determined by measuring its length, L, breadth, B, height, H and then multiplying the results together. In equation form, the volume, V of the material can be expressed thus

$$V = LBH \tag{2}$$

Now, if B and H are same as L, the material is said to be in the form of a cube. Therefore,

$$V = L^3 \tag{3}$$

But for a material that is in the form of a cylinder of uniform diameter, the area, A of one of its circular surfaces and the separation, h between those surfaces are used to compute its volume using the relation

$$V = Ah \tag{4}$$

If d is the diameter of either circular surface of such material, then

$$A = \frac{\pi d^2}{4} \tag{5}$$

and so,

$$V = \frac{\pi d^2 h}{4} \tag{6}$$

In addition, there are other forms of regular shape in which material can be, namely, cone, pyramid, prism, etc and their volumes can be computed as described by Tuttuh-Adegun and Adegoke 2011, Ndupu et al 2000 and also stated in several textbooks as well as mathematical tables.

Water Displacement Method

This method can be used to measure the volume of non-porous solids of whatever shape. It solely relies on the fact that water remains incompressible for all practical purposes and as such, whenever a solid is made to enter water that is held in a container, the water gets displaced so as to make room for the solid. Also, when the solid is fully immersed in the water, it displaces the volume of water that is exactly equal to its own volume. This observation is simply Archimedes' Principle in action (Okeke et al 2011).

In the present application of the method, a graduated measuring cylinder is first filled with water to a reasonable volume mark and then the material whose volume is to be measured is made to submerge in the water. To obtain the volume of the material, the difference between the volume of water before and the one after the immersion is calculated. In expressing mathematically to show that the volume, V of the material is the same as that of the water displaced, it can be stated thus

$$V = V_f - V_i \tag{7}$$

Where

V_i = volume of water in the cylinder before immersion of the material

V_f = volume of water in the cylinder after immersion of the material.

As earlier stated in this work, it is unsuitable to use this traditional water displacement method if the material is porous because water has a high tendency to affect a porous material when it is in contact with it.

Modified Water Displacement Method

In this research work, the modification of the traditional water displacement technique described above is in terms of two aspects. One of such is waterproofing of the porous material to be used so that it can be immersed in the water without being affected adversely. For effective waterproofing, sealing of the porous material is necessary. Another aspect is the determination of the volume of the material based on the internal diameter of the cylinder used and the displacement of the water when the sealed material is completely immersed in it. Now, using equation (1) above, if a sealant of density ρ_s is used to seal a porous material of mass, m, the volume, VS of the sealant can be calculated thus

$$V_s = \frac{W - m}{\rho_s} \quad (8)$$

where W is the mass of the sealed material used. Also, when the water held in the cylinder is displaced as a result of fully immersing the sealed material in it, the only place for the water to go is up. So, by considering the fact that any liquid does not have a fixed shape but it takes that of its container, it is crystal clear that the water displaced by the sealed material, in this case, will be cylindrical in shape.

Therefore, if the cylinder used is of external diameter, D, and uniform thickness, t, then its internal diameter, di, which of course is same as the diameter of the cylindrically displaced water can be obtained using the relation

$$d_i = (D - 2t) \quad (9)$$

By substituting d_i for d and also, the displacement, x of the water for h in equation (6) above, the volume, V_m of the sealed material can be expressed as

$$V_m = \frac{\pi (D - 2t)^2 x}{4} \quad (10)$$

Hence, the bulk volume, V, of the porous material can be deduced using the equation

$$V = V_m - V_s \quad (11)$$

and then the corresponding bulk density can be computed using equation (1) above.

Alternatively, the bulk density, in this case, can be determined by substituting equation (11) into equation (1) and then using the resulting equation, which is

$$\rho = \frac{m}{V_m - V_s} \quad (12)$$

EXPERIMENTAL DETAILS

Materials and their Collection

The materials used in this work were asbestos ceiling board, chalk, cardboard paper, clay (compacted), gypsum board, pure paraffin wax, and water. While the pure paraffin wax, cardboard paper, chalk and gypsum board were bought directly from their respective marketers, the asbestos ceiling board was obtained from a building construction site and the clay was got from a low lying area with intermittent water flows. All the materials listed above were collected within Uyo metropolis in Akwa Ibom state situated between latitude 403211 and 503311 North and longitude 703511 and 802511 East.

Equipment and their Uses

In this work, the following equipment was used as indicated:

Glass cylinder of uniform thickness – for holding the water.

Digital thermometer – for measuring temperature using a k-type probe with the sensor.

Analytical balance – for weighing masses.

Digital Vernier calipers – for measuring the external diameter of the cylinder used.

Knife – for cutting and trimming the porous materials into the test samples of regular shape.

Digital micrometer screw gauge – for measuring the thickness of the glass cylinder.

Coffee can – for holding the paraffin wax during melting.

Double boiler – used as a means of melting the paraffin wax.

Uniform meter rule – for linear measurement of test samples dimensions as well as the displacement of water contained in the cylinder.

Preparation of the Test Samples

All the porous materials gathered for this research work were subjected to continuous sun-drying and weighing until no change in mass was observed in each case. This was necessary in order to ensure that the test samples contained no moisture. The materials were then cut into three test samples, each, of regular shape. Also, one sample, being the fourth in each case was shaped for the standard test in order to determine the reference bulk density value. Some of the samples are shown in figure 1 below.

Figure 1: Test samples of the dry porous materials

Method

The mass of each test sample was measured with the analytical balance (METTLER TOLEDO model PL203, d = 0.001g) and the bulk volume of each meant for trial runs was determined by geometry method, after which their corresponding bulk density was computed according to equation (1).
Thereafter, the paraffin wax was put in the coffee can and subjected to melting by means of the double boiler. A coat of the melted wax was applied to the samples with the brush and when it has hardened, another coat was applied to completely seal and make the sample water-resistant. The glass cylinder was filled with water to a reasonable level and the water temperature was measured. Each of the sealed samples was made to completely immerse in the water with care to ensure that the water did not splash onto the sides of the cylinder, and also, the amount of water displacement observed in each case was measured. The measurements taken for this modified water displacement technique were used to compute the bulk volumes of the samples by applying equations (8), (10) and (11). Also, equation 1 was applied in determining the respective bulk densities after which the results were tabulated alongside the reference bulk density values obtained in accordance with (ASTM D6683-14, 2014). The results were analyzed statistically and graphically.

RESULTS AND ANALYSIS

The results and analysis are shown in Tables 1 and 2, and also in figure 2.

Table 1: Data obtained for the use of Geometry method and the Modified Water Displacement Method to determine the bulk densities of the materials

Materials	**Trials**	**m(g)**	**Geometry Method**			**Modified Water Displacement Method**				
			L (cm)	**B (cm)**	**H (cm)**	***W* (g)**	***x* (cm)**	**D (cm)**	**t (cm)**	ρ_s **(gcm^{-3})**
Asbestos Ceiling board	1	2.270	2.7	2.2	0.3	3.930	0.4			
	2	3.301	2.9	2.3	0.3	4.869	0.5			
	3	5.512	4.5	2.6	0.3	7.335	0.7			
Cardboard Paper	1	0.504	2.8	2.2	0.1	1.109	0.2	3.20	0.132	0.9
	2	0.722	3.0	2.4	0.1	1.607	0.3			
	3	1.013	4.9	2.6	0.1	2.216	0.4			

Chalk	1	1.302	2.6	0.8	0.7	1.910	0.2
	2	1.820	3.2	0.8	0.7	2.724	0.3
	3	2.811	5.2	0.8	0.7	4.025	0.4
Clay (Compacted)	1	2.202	1.4	1.4	0.6	3.115	0.3
	2	3.201	1.4	1.3	1.0	4.724	0.5
	3	8.000	1.9	1.8	1.3	11.962	1.3
Gypsum board	1	3.345	1.7	1.3	0.7	3.840	0.3
	2	3.838	1.9	1.4	0.7	5.380	0.5
	3	5.694	2.2	1.7	0.7	7.733	0.7

Table 2: Statistics of Bulk Density values obtained at room temperature

Materials	**Test methods used and Bulk Density values in gcm^{-3}**								
	ASTM D6683-14 (Ref. Values)	**Geometry Method**				**Modified Water Displacement Method**			
		1	**2**	**3**	**Mean value**	**1**	**2**	**3**	**Mean value**
Asbestos ceiling board	1.985	1.526	1.650	1.570	1.582±0.041	1.994	2.008	2.031	2.011±0.012
Cardboard paper	0.701	0.818	1.003	0.795	0.872±0.069	0.739	0.689	0.739	0.722±0.017
Chalk	1.902	0.894	1.016	0.965	0.958±0.041	1.920	1.772	2.068	1.920±0.099
Clay (Compacted)	1.852	1.872	1.759	1.799	1.810±0.038	2.165	1.890	1.818	1.958±0.116
Gypsum board	2.348	2.162	2.061	2.175	2.133±0.034	2.258	2.294	2.302	2.285±0.015

DISCUSSIONS

In this work, the measurement results for determination of bulk densities by geometry method and the modified water displacement method are presented in table 1, while the calculated bulk density values are presented in table 2 with their corresponding reference values obtained according to ASTM D6683-14. Also, the graphical analysis of the results is shown in figure 2. From table 1, it can be seen that for each of the materials, the mass and water displacement increase with the volume of the samples used in the trial runs. This, obviously, is due to the fact that the samples used were cut from a particular material and as such, they were made up of same matter at the same degree of compaction during preparation. In this case, such observation signals that the mass of a particular material is proportional to its volume, and it can be expressed mathematically by transforming a well and widely used formula for density calculation presented in this work as equation (1).

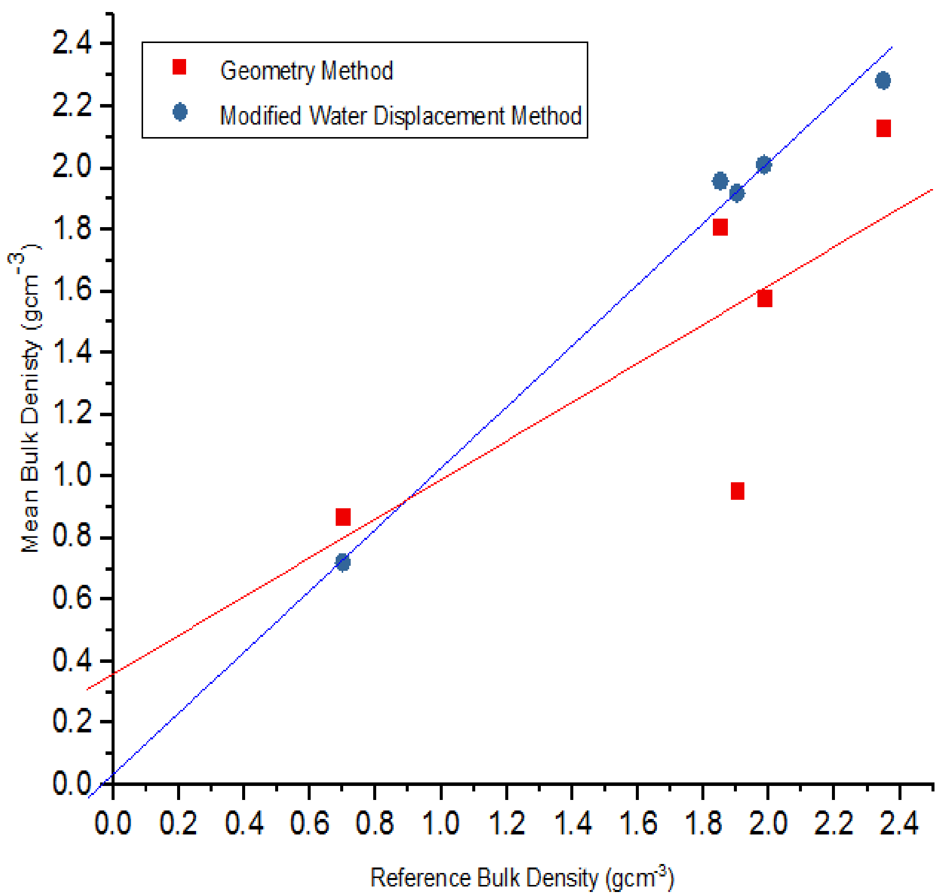

Figure 2: Mean Bulk Density values versus their corresponding reference bulk density values

Also, based on the results shown in table 2, a quantitative comparison between the mean bulk density values and the corresponding reference values shows that the percentage error associated with the use of geometry method ranges from 2.3% with cardboard paper to 49.6% with chalk. However, the reverse is the case with the use of the modified water displacement method as the lowest error value is 0.9% with chalk and the highest error value is 5.7% with the cardboard paper. This shows that the modified water displacement method yields results of higher accuracy than the geometry method. The reason for the limited accuracy in the mean bulk densities determined by the geometry method can be understood to be that the surfaces of the porous materials are imperfect. This imperfection may be as a result of the materials having surface irregularities, small fractures and fissures, etc which communicate with the materials' surfaces and are isolated within the structure, thus making the bulk volumes of the materials determined from linear measurements of their length, breadth and height to have limited accuracy and consequently affect the bulk density values computed.

Statistically, the Pearson's product moment correlation coefficient calculated between the mean bulk densities of the test materials and their corresponding reference values at 0.05 level of significance with degree of freedom of 3 gives 0.7430 and 0.9955 for the use of geometry method and modified water displacement method respectively whereas, the critical value of the coefficient is 0.8783. Since the correlation coefficient value is -1.0 for a perfect negative linear relationship and 1.0 for a perfect positive linear relationship, it means that the results obtained by the modified water displacement method have a greater degree of similarity than those obtained by the geometry, with the reference values. In other words, the critical value of the correlation coefficient being greater than 0.7430 but less than 0.9955 implies that even though both methods relate positively with the standard test method adopted in this work, the modified water displacement method is more reliable than the geometry method.

Moreso, figure 2 shows the data plots illustrating the regression of the geometry method and modified water displacement method as experimental methods on the ASTM D6683-14 used as a standard method, by plotting the mean bulk densities of the test materials against their reference values and fitting the lines using the least square method. For the geometry method, the fitted line has a slope of 0.6511 with an angle of 33.1^0 whereas the plot for the modified water displacement method has a line with a slope of 0.9736 and angle of 44.2^0. By carefully studying the graphical representation of data of this nature, it is very obvious that when there is a positive correlation between two graphically compared data sets without bias, the fitted line will have zero intercepts with regression line slope of one thereby making the slope angle to be 45^0. In such a case, the pairs of data are said to be ideally similar. This concept was similarly advanced by Kelly 1987 as a method of comparison.

In this research work, the low slope value and angle indicate limited accuracy in the results obtained by the geometry method and its computed mean bulk density values can be seen to diverge far off the line confirming their wide deviation from the reference values whereas the mean bulk density values, determined using the modified water displacement method, that does not fall on the line are very close to it. Also, it is clearly observed that the slope value and angle associated with the modified water displacement method only are approximately 1 and almost 45^0 respectively. This shows that unlike the results obtained by the use of geometry method, those obtained by the modified water displacement method are in close agreement with the reference values thereby implying that the modified water displacement method is almost similar to and as effective as the ASTM D6683-14 used in this work. This principle of analyzing the similarity between the two methods is supported by the work of Miller & Miller 2010 in comparing similarities between two methods.

CONCLUSION

The results of mean bulk densities obtained by geometry method in this work yielded Pearson's correlation coefficient of 0.7430 which was observed to be lower than the critical value of 0.8783 at 0.05 level of significance with degree of freedom of 3 whereas the mean bulk density values determined by using the modified water displacement method resulted in the correlation coefficient of 0.9955 when, in both cases, the results were statistically correlated with the reference bulk density values obtained in accordance with ASTM D6683-14. Also, from the graphical analysis of the results, the slope values of 0.6511 and 0.9736 as well as slope angles of 33.1^0 and 44.2^0 were obtained when comparing the geometry method and modified water displacement method, respectively, with the ASTM D6683-14.

SUMMARY

Generally, it was found that although the two methods are common, cost-effective, and can be used to determine the bulk density of a porous material, the modified water displacement method is more effective and far better than the geometry method in terms of accuracy and reliability. Also, even if the available glass cylinder is not graduated, the modified water displacement method can be employed to obtain a bulk density value that is valid, reliable and very high in accuracy

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