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Home-made vacuum system for calculating the apparent density of solid materials

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ABSTRACT

Home-made vacuum system was set up for measuring the apparent density of solid materials or more accurately the volume of solid materials. The volume of samples was calculated based on the method of gas displacement and the inversely proportional relationship between the absolute pressure and volume of a gas known as Boyle's Law, $pV=k$. Nitrogen gas was used as a probe and the measurement was done at room temperature and at low pressure in order to minimize the effect of the compressibility factor of the gas. The system was calibrated by stainless steel ball with the error was less than 5%. The apparent density of hollow stainless steel (non-porous), rice (irregularly shaped) and Clorets sweet (regularly shaped) were 7.61, 1.60 and 1.23 *g/mL*, respectively. Whereas the apparent density of porous powdered materials were 1.34, 1.26 and 1.67 *g/mL* for Pt/SO_4^{2-}-Al_2O_3, WO_3-ZrO_2 and Portland Cement, respectively. The experimental error for measuring the solid volume was less than 5%.

| Apparent density | Vacuum system | Pt/SO_4^{2-}-Al_2O_3 | WO_3-ZrO_2 | Portland cement | Stainless steel | Rice | Sweet |

1. INTRODUCTION

Density is a physical property of matter, as each element and compound has a unique density associated with it [1]. Archimedes had used the concept of density to expose the fraud [2]. Density defined in a qualitative manner as the measure of the relative "heaviness" of objects with a constant volume. The more mass an object contains in a given space, the denser it is. It is important to remember, though, that this relationship is not just about how closely packed together the atoms of an element or molecules of a compound are. Density is also affected by the atomic mass of an element or compound. Since different substances have different densities, density measurements are a useful for identifying the properties of substances. In some cases density is expressed as the dimensionless quantities specific gravity (*SG*) or relative density (*RD*), in which case it is expressed in multiples of the density of some other standard material, usually water or air. In a chemical reactor design, density can be related with space velocity (τ) where it represents the relation between volumetric flow and reactor volume or catalyst bed volume [3].

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The density of fluids is easier to be measured compared to the solids due to the easier volume measurement of fluids. Glass pycnometer is a device usually used to measure the density of fluids accurately by using a reference to an appropriate working fluid, such as water or mercury, using an analytical balance. However, the density measurement of solids was lagging behind because of their difficulties in the measuring of volume of sample especially for porous or non-porous, regularly or irregularly shaped, powdered, monolithic, and/or granular materials.

In this study, we have set up a simple vacuum system for measuring the volume of solid shapeless material such as porous, non porous, powdered, regularly and irregularly materials. The volume of solid shapeless sample was calculated based on the displacement of gas and the Boyle's Law in which the volume of system is inversely proportional to the absolute pressure of system, $pV=k$. Then, the apparent density, ρ of the sample can be determined by its mass per unit volume of samples as expressed in Eq(1),

$$\rho = M / V \qquad (1)$$

where, M and V are mass (g) and volume (mL) of sample respectively.

2. EXPERIMENTAL

2.1 Porous powdered samples

Pt/SO_4^{2-}-Al_2O_3 and WO_3-ZrO_2 porous powdered samples were prepared as follows.

The SO_4^{2-}-Al_2O_3 was prepared by impregnation of the $Al(OH)_3$ (Merck) with 1N H_2SO_4 aqueous solution (Merck) followed by filtration, drying at 383 *K* overnight and calcination at 823 *K* for 3 *h* in air [4]. Pt/SO_4^{2-}-Al_2O_3 was prepared by impregnation of SO_4^{2-}-Al_2O_3 with $H_2PtCl_6{\cdot}6H_2O$ aqueous solution (Merck) at 353 K, followed by drying at 383 *K* overnight and calcination at 723 *K* for 3 *h* in air. The content of Pt was 0.5 wt%.

The WO_3-ZrO_2 sample was prepared by impregnation of $Zr(OH)_4$ with aqueous solution of ammonium metatungstate $(NH_4)_6[H_2W_{12}O_{40}]$, followed by drying at 383 *K* overnight and calcinations at 1093 *K* for 3 *h* in air [5]. The content of WO_3 was 13 wt%. Zirconium hydroxide $Zr(OH)_4$ was prepared by titration of zirconium oxychloride ($ZrOCl_2{\cdot}8H_2O$) (Wako) with ammonia (NH_3) solution (Merck) [5]. 50 *g* of zirconium oxychloride was dissolved with 2.5 *L* of doubly distilled water. Then, 2.8 % of ammonia solution was added dropwise by titration method up to pH 8 at 353 *K* under vigorous stirring. White colloidal precipitate was formed at pH 7. Then, 28 % of ammonia solution was added again dropwise by titration method up to pH 10 at 353 *K* with vigorous stirring. Then the precipitated hydrogel was aged for 6 *h* at this temperature. The solution was decanted with deionized-distilled water at room temperature until the pH of solution becomes 7. The zirconium hydroxide precipitate obtained was filtered and dried at 383 *K* overnight in air. Ammonium metatungstate solution was prepared by the reaction of tungsten oxide (Wako) with ammomiun solution (Merck) at 353 *K*.

Pt/SO_4^{2-}-Al_2O_3 WO_3-ZrO_2 Portland Cement

Figure 1. The picture of powdered samples

The apparent density of commercial Portland cement was also measured in this experiment. The chemical composition of commercial Portland cement is 50–70% C_3S, 15–30% C_2S, 5–10% C_3A, 5–15% C_4AF, and 3–8% other additives or minerals (such as oxides of calcium and magnesium). Where C, S, A and F mean for CaO,SiO_2, Al_2O_3 and Fe_2O_3, respectively [6]. The standard density of Portland cement is 1.506 *g/mL* [7]. Figure 1 illustrates the picture of powder samples.

Stainless steel Faiza rice Clorets sweet

Figure 2. The picture of non-porous, irregularly and regularly shaped samples

2.2 Non-porous regularly and iregularly shaped solid samples

Commercial non-porous hollow stainless steel grade 316 (Swagelok, SS-T2-S-035-6ME), irregularly shaped Faiza rice (Grade A) and regularly shaped Clorets sweet (Cadbury Adams Factory No.: CICOT.HL87A4060330248) were used as solid samples in this experiment. Figure 2 illustrates the picture of solid samples.

The chemical composition of stainless steel grade 316 is Fe, <0.03% C, 16-18.5% Cr, 10-14% Ni, 2-3% Mo, <2% Mn, <1% Si, <0.045% P, <0.03% S with the standard density of 8.00 *g/mL* [8].

2.3 Characterization of catalysts

Bruker AXS D8 Automatic Powder Diffractometer was used to determine the crystalline structure of powder samples. The specific surface area was determined with Quantachrome Autosorb-1 at 77 *K*. Prior to the analysis the sample was evacuated at 573 K for 3 *h* due to the removal of moisture or any contaminant in the pores.

2.4 Home-made vacuum system

Figure 3 shows the schematic diagram of home-made vacuum system for measuring the volume of solid materials. The system consists of 2 chambers, first chamber is to hold the sample and called as a sample holder and the second chamber is an internal volume of the system (consists of vacuum line and N_2 gas storage) and called as a reference volume. The system was equipped with an Edward Diffusion Pump which is able to evacuate the system reached 10^{-6} *Torr*, furnace, temperature controller, pressure transducer, Ecoder and computer installed with Edaq software for monitoring the pressure change in the system.

The valumes of both chambers were calibrated with Ø2mm stainless steel ball (V=0.5236 *mL*). The volumes of first and second chambers were 11.20±0.05 and 143.17±0.03 *mL*, respectively.

2.5 Experimental procedure

The measurement of volume of solid shapeless materials was done at room temperature and at low presure in order to minimize the effect of compressibility factor of nitrogen gas.

The glass vacuum line (2) and sample holder containing a sample (3) were evacuated to 0.00 *Torr*. Then, glass stopcock (1) and stopcock between vacuum line and sample holder were closed, nitrogen gas storage with 200 *Torr* of gas was opened, then glass stopcock between vacuum line and sample holder containing sample was then opened and the pressure change of system was recorded. Based on the Boyle's Law ($pV=k$), the valume of sample can be measured by Eq(2).

$$V_s=V_c + \{V_r/(1-P_1/P_2)\} \quad (2)$$

where V_s is the sample volume, V_c is the volume of the empty sample chamber, V_r is the volume of the reference volume, P_1 is the first pressure and P_2 is the second pressure after expansion of the gas into the combined volumes of sample chamber and reference chamber. The apparent density of sample can be determined as a ratio of mass to volume of sample.

For the porous powdered sample, the sample was subjected to outgassing at 573 *K* for 3 *h* in order to remove the moisture content and any contaminant in the pores.

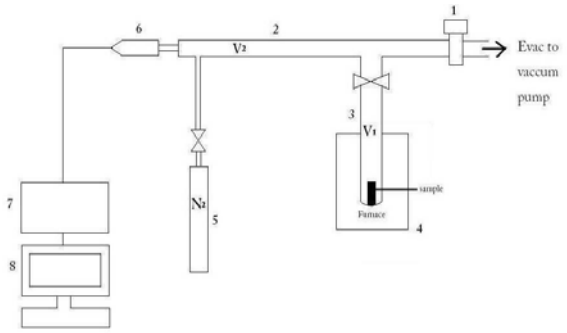

Figure 3. Schematic diagram of home-made vacuum system. (1) Glass stopcock, (2) Glass vacuum line, (3) Sample holder, (4) Furnace, (5) N_2 gas storage, (6) Pressure transducer, (7) Ecoder, (8) Computer with Edaq software.

3 RESULTS AND DISCUSSION

Figure 4 shows the diffractograms of Pt/SO_4^{2-}-Al_2O_3 and WO_3-ZrO_2 samples. For Pt/SO_4^{2-}-Al_2O_3 samples, the addition of sulfate ion and platinum on γ-Al_2O_3 did not change the structute of γ-Al_2O_3 [9,10]. The intense peaks at about 2θ=38, 46 and 67° coresponding to γ-Al_2O_3 (110), (111) and (211) were observed clearly. The surface area of Pt/SO_4^{2-}-Al_2O_3 was 163 m^2/g. XRD patterns of WO_3-ZrO_2 indicated the presence of tetragonal and monoclinic phases of zirconia. The intense peaks corresponding to monoclinic phase of zirconia was observed at about 2θ=28 and 32° [11,12]. While the peaks corresponding to tetragonal phase of zirconia was observed at about 2θ=30° [13]. The surface area of WO_3-ZrO_2 was 51 m^2/g.

Table 1 lists the density of porous powdered samples. The density of Pt/SO_4^{2-}-Al_2O_3 and WO_3-ZrO_2 are almost similar and they are higher than the density of water due to the metal contents such as Pt, Al, W and Zr on the sample [14]. The reference density of Pt free SO_4^{2-}-Al_2O_3 was 1.29 *g/mL* [7]. The presence of Pt increased slightly the density of the sample. The deviation for Pt/SO_4^{2-}-Al_2O_3 from the density of SO_4^{2-}-Al_2O_3 was in an acceptable range of about 3.8%. The density of commercial Portland cement was 1.67 *g/mL* which is slightly higher than that of the reference density of 1.506 *g/mL* [7]. A lot of metal contents in the cement contributed to the higher density compared to that of Pt/SO_4^{2-}-Al_2O_3 and WO_3-ZrO_2 samples.

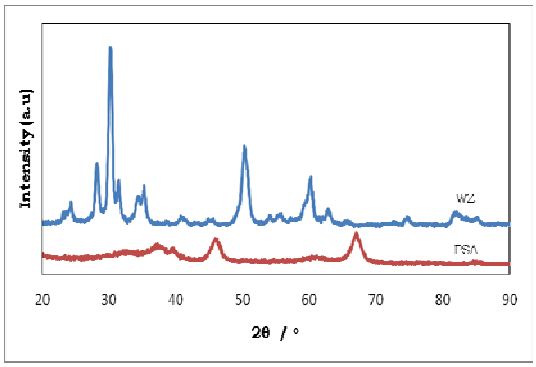

Figure 4. The XRD patterns of calcined Pt/SO_4^{2-}-Al_2O_3 (PSA) and WO_3-ZrO_2 (WZ).

The differences of the density values in this experiment with the reference values may be caused by the differences in the pretreatment technique and/or the moisture content in the pore of sample. Over all the error for the measuring of powder material in this experiment was less than 10%.

Table 1. Density of porous powdered samples

Sample	Density (*g/mL*)
Pt/SO_4^{2-}-Al_2O_3	1.34 ± 0.08
WO_3-ZrO_2	1.26 ± 0.07
Portland Cement	1.67 ± 0.09

Table 2. Density of non-porous, irregularly and regularly shaped samples

Sample	Density (*g/mL*)
Stainless Steel	7.61 ± 0.02
Rice	1.60 ± 0.04
Clorets Sweet	1.23 ± 0.05

Table 2 lists the density of non-porous, irregularly and regularly shaped samples. The density of non-porous hollow stainless steel grade 316 was 7.61 *g/mL*. This value

is lower than that of the reference of 8.00 *g/mL* [8]. The difference in the value may be caused by the experimental errors. The densities of irregularly shaped rice and regularly shaped Clorets sweet were 1.6 and 1.23 *g/mL*, respectively. The densities of rice and sweet are lower than that of the stainless steel due to the metal content and compact arrangement of metal in the stainless steel.

4. CONCLUSION

Vacuum system to measure the volume of solid materials has been proposed based on the Boyle's Law. The apparent density of solid materials can be determined by ratio of mass to the volume of the sample. In this experiment, we reported the apparent density of Pt/SO_4^{2-}-Al_2O_3, WO_3-ZrO_2 and Portland cement were 1.34, 1.26 and 1.67 *g/mL*, respectively. While the apparent density of stainless steel, rice and Clorets sweet were 7.61, 1.60 and 1.23 *g/mL*, respectively. The experimental error in this experiment was less than 10% for all samples.

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