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Measuring surface-connected porosity of complete 3d printed structures using a custom pycnometer

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Abstract: 3D Printed (3DP) medical parts continue to be adopted for a variety of uses. Part quality is a continuing concern, with one aspect being cleanliness and the ability to be effectively cleaned. Surface roughness and surface porosity are properties that make cleaning more difficult. A method of using pycnometry is described to measure porosity in complete 3DP parts. Pycnometry uses changes in volume and pressure to calculate the volume of a sample. The system is able to identify small variations in sample volume, and could be used to compare the actual part volume with the expected volume. While pycnometry is a well-established technique, regulated 3D printed medical devices pose a challenge because commercial pycnometry systems are designed to evaluate coupons, rather than complete commercial products.

I. Introduction

As 3D printing (3DP) continues to infiltrate the medical market, questions of cleanliness become more important for finished parts. Indeed, the United States Food and Drug Administration (FDA) has placed an emphasis on this topic, specifying improvements in quality assessment in their strategic plan for regulatory science [1]. Unintended surface-connected porosity in a 3D print may trap body fluids and microorganisms constituting a safety hazard. FDA does not clear or approve medical device components, but only final products in their to-be-marketed form. In particular, 3DP coupons may not reflect performance of an entire 3DP device. Analytical methods such as micro-computed tomography or liquid immersion are often unacceptable because of potential errors from sampling assumptions or retention of material that renders the tested device medically unusable. Our goal was to evaluate a validated method of surface-connected porosity assessment that could be applied to a complete 3D printed commercial medical device to which a patient would be exposed.

I.I. Pycnometry

Pycnometry is a quantitative, non-destructive means of assessing the volume of an object that does not require immersion. Pycnometry has been used previously with 3DP parts [2-3], but with the goal of quantifying the accurate *production* of porous features, not the presence of non-porosity as a means of quality assurance (QA).

The basis of pycnometry is Boyle's Law, which itself is derived from the Ideal Gas Law. Boyle's Law, shown in (1), relates the pressure and volume of a closed system.

$$P_1 V_1 = P_2 V_2 \quad (1)$$

A more thorough review of the theory and equations used for pycnometry can be found elsewhere [4]. Briefly, a sample chamber of volume V_c that contains a sample of volume V_p (typically unknown) is pressurized to a pressure P_1. An additional volume V_a is then introduced to the closed system, thereby reducing pressure to P_2. By applying Boyle's Law, the equation to determine sample volume is shown in (2):

$$V_p = V_c + \frac{V_a}{\left(1-\frac{P_1}{P_2}\right)} \quad (2)$$

Several assumptions must be made in utilizing these elementary equations. The supply gas is assumed to be ideal. Environmental conditions must remain static. All chambers and tubing must be rigid and not expand under pressure. Such compliance wound introduce error into the calculation.

As indicated above, the size of the chamber volume (V_c)should be very close to volume of the part being tested (V_p) for maximum accuracy. Furthermore, an optimal added volume (V_a) may be predicted for each experimental set-up. Commercial pycnometry devices generally do not allow for chamber volume adjustments, or are too small to test most complete 3D printed medical devices.

I.II. Ideal versus actual volume

The surface tessellation file (STL) enables prediction of a 3DP volume within an arbitrary level of resolution. The physical volume of the 3DP part depends on the additive manufacturing method used, printer quality, materials, and also includes surface connected porosity as an error in an otherwise well-controlled manufacturing process. The difference between $V_{p,actual}$ and $V_{p,ideal}$ can yield insight to the degree of surface-connected porosity present in the analyzed part. However, any internal voids not in fluid communication with surrounding space are not reflected in pycnometry.

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II. Material and methods

The tested objects used in the experiments were a custom-fit cast (ActivArmor Inc, Pueblo Colorado, USA) made from Acrylonitrile butadiene styrene (ABS) using a fused deposition modeling (FDM) process. The outer dimensions are approximately 150mm x 50mm x 40mm (LxWxH)

The pycnometric measurement system is shown schematically in Fig. 1.

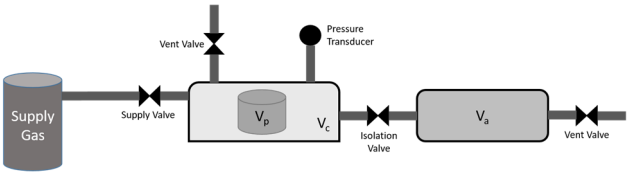

Figure 1: Pycnometry apparatus schematic

For the purposes of this investigation, a syringe pump was used as a pressure source. The gas used within the chambers was atmospheric air. Initial tests were performed by modifying the overall volume of V_c, and recording the subsequent different in P_2. Calibration testing was conducted over a range of precisely controlled changes in V_c before and after opening the system to V_a. In this way, pressure changes from P_1 to P_2 mimicked the introduction of a 3D printed part of known volume into V_c.

In order to minimize the difference between V_c and V_p, the sample chamber was fabricated to closely conform to the 3D printed ActivArmor™ device being tested. The chamber used a thermoformed sheet (Compac Mini, Formech Inc, Harpenden, UK) contoured around the test object. A cast acrylic epoxy shell augmented rigidity of the thermoformed sheet. The two halves of the chamber were sealed with silicone caulk and bolted together to achieve a gas-tight seal. V_c, measured without a 3D print was measured in our calibrated set-up.

Next, after equalizing chambers to ambient pressure P_0, the test article was placed into the conformal chamber V_c of known volume, then pressurized to P_1.The supply valve was closed, and the valve between V_c and V_a opened to measure the drop in system pressure to P_2 (AMS5812, Analog Microelectronics GmbH, Mainz, Germany) in the experimental configuration.

III. Results and discussion

Small changes to the expected part volume, e.g. 0.5 mL, were represented by easily discernible changes in pressure when V_c was opened to V_a as shown in Fig. 2.

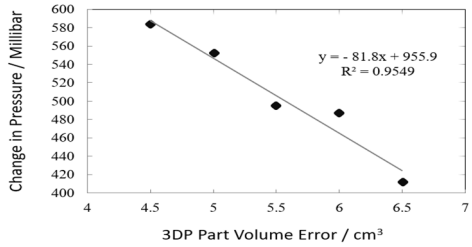

Figure 2: Relationship of part volume to pressure drop.

In order to demonstrate the reproducibility of the experimental pressure measurements, several trials were repeated with each 3D print at the same V_c, P_1, and V_a. Fig. 4 illustrates that repeated measurements of the yielded quantitatively comparable transitions from P_1 to P_2.

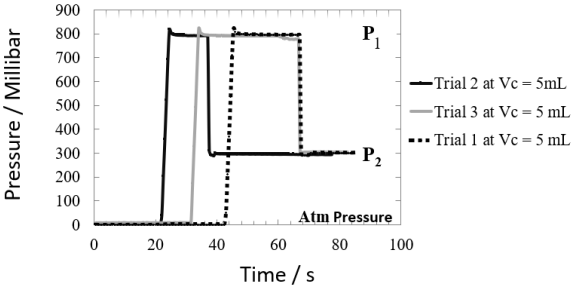

Figure 3: Repeated measurement of the same sample had identical transitions in pressure.

Pressure transitions are shown at different points on the time base in Figure 4 because the valve opening V_a to V_c was thrown manually at slightly different times after automated data capture had begun. Additionally, small transients in overshoot were likely a consequence of the elastic stretch of the pressure tubing.

IV. Conclusions

Our findings suggest that gas pycnometry using a test chamber specifically designed for certain fully-finished 3D printed medical devices may provide a simple method to characterize surface-connected porosity. In particular, this method is not unduly burdensome so that every complete 3D printed part to which a patient's body may be exposed may be evaluated prior to commercial release of each individual medical device. Use of customized gas pycnometry may be considered when approaching regulatory review questions regarding potential for biological intrusion into 3D printed medical devices in contact with a patient's body.

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