# Advanced Method for Void Fraction Evaluation of Natural Fiber Composites using Micro-Ct Technology

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# ADVANCED METHOD FOR VOIDFRACTION EVALUATION OF NATURAL FIBER COMPOSITES USING MICRO-CT TECHNOLOGY

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## ABSTRACT

Natural fiber reinforced composites have been gaining a lot attention in the past couple of decades and they have been developing to be used in more advanced engineering and structural applications. Fiber volume fraction is one of the most important properties when working with composite processing and engineering design. Mechanical properties of composites materials are in direct proportion with their fiber volume fraction. There are challenges and errors involved with calculation of fiber volume fraction and void fraction in composite materials, especially when working with natural fiber reinforced composites. In this paper, micro-CT technology was used to acquire 3-D scans of specimens from different composites samples. A MATLAB code was developed using image processing functions to evaluate the images extracted from micro-CT scans, and void percentage of each sample was determined. Void fraction measurements of eight composite samples were analyzed and results were compared against the calculated values. Results of this study suggest that the micro-CT technology can be used as a reliable tool for evaluating composite materials and calculations of void fractions.

## 1. INTRODUCTION

Natural fibers such as kenaf, hemp, flax, jute, sisal and nettle have been the center of attention as natural reinforcement in composite materials for the past two decades due to their superior advantages. Use of natural fibers as reinforcement in polymer composites instead of synthetic or mineral fibers, provides competitive strength to weight ratios. In addition, replacing synthetic fibers with natural fibers contributes to improvements in environmental performance of end products [1, 2]. Researchers have performed various studies on natural fiber technology [3-5], their use as reinforcement in polymer composites [6-13], as well the service life assessment and environmental impacts of bio-based composites [14, 15].

### 1.1 Flax fiber

Flax is a type of crop fiber which is grown both for fiber (linen) and for seed oil (linseed). Flax is a type of multicellular fiber in which its properties are defined by physical, mechanical and chemical properties of the morphological constituents such as cellulose, hemicellulose, lignin and pectin.



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Figure 1 shows the structure of flax fiber cell. There are several layers in a single fiber [16-19]. The primary wall, which contains both cellulose and hemicellulose, is the first layer dispositioning during plant growth [20]. The secondary wall includes three layers and consists of helically wound highly crystalline cellulose chains called micro-fibrils. These micro-fibrils are made up of 30 to 100 cellulose molecule chains which are oriented with approximately a 10º angle. The angle of micro-fibrils in relation to the axis of the fiber will dictate the rigidity of the fiber [20]. Micro-fibrils of cellulose are held together by amorphous regions consisting of hemicellulose and lignin. The secondary wall of the fiber determines 70% of the fiber young's modulus [21].

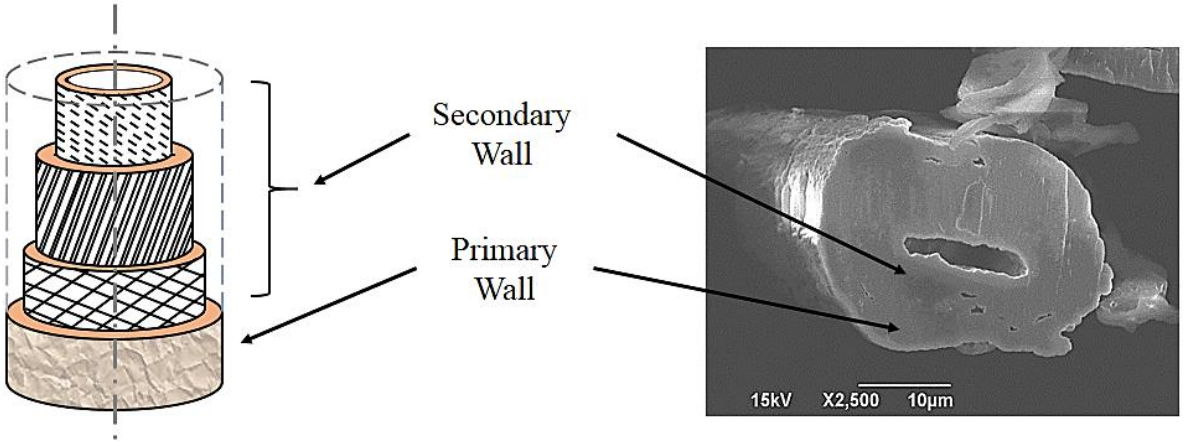


Figure 1. The structure of flax fiber cell [1].

Cellulose is a natural polymer consisting of D-anhydro-glucose, $C_6H_{11}O_5$, joined by β-1, 4-glycosidic chains at $C_1$ and $C_4$ locations [22] and there are three hydroxyl groups attached to every repeating cellulose unit. Presence of three hydroxyl groups in each repeating unit will make cellulose a hydrophilic molecule [1, 21, 23].

### 1.2 Void volume fraction

For design purposes and to compare properties of two laminates, one should know the fiber volume fraction of the composite. Mechanical properties of composite materials are highly sensitive to fiber volume fraction [24-26]. Fiber volume fraction is defined as [27]:

$$V_f = Fiber\ volume\ fraction = \frac{volume\ of\ fiber}{volume\ of\ composite} \quad (1)$$

Consequently, the void content of composite can be found[27]:

$$V_v = Void\ volume\ fraction = 1 - V_f - V_m = \frac{volume\ of\ voids}{volume\ of\ composite} \quad (2)$$

where $V_m$ is the matrix volume ratio. The void fraction of composite can be found by comparing experimental fiber volume fraction and theoretical fiber volume fraction. The theoretical fiber volume fraction is calculated by[26]:

$$V_f = \frac{\rho_f - \rho_m}{\rho_c - \rho_m} \quad (3)$$

where, $\rho_f$, $\rho_c$and $\rho_m$ are the density of fiber, composite and matrix, respectively. The experimental fiber volume fraction is calculated by[26]:

$$V_f = \frac{w_f}{w_c}\frac{\rho_c}{\rho_f} \quad (4)$$

where $w_f$ and $w_c$ are the weights of fiber and composite, respectively.

Due to the discussed structure, surface morphology, and hydrophilic nature of natural fibers, accurately measuring the density of natural fibers challenging. In this paper an alternative method is suggested using the latest technology to find void fraction in composite materials. In this method, 3D images of specimens from composite samples are acquired using micro-CT scans. 3D scans are sliced in layers with 0.01 mm thickness and the void percent of each layer is measured using MATLAB® image processing functions. The results are compared against void fractions calculated from conventional methods using Equations 1-4.

## 2. EXPERIMENTATION

### 2.1 Materials and methods

Five different types of flax fiber were used in this study. Four types of linen flax, farmed and harvested by the University of Saskatchewan, Saskatoon, SK, Canada. Shive (i.e. woody core of the flax stalk) was removed by passing the fiber through a pilot line eight times at Biolin Research, Inc., Saskatoon, SK, Canada. Three different mechanical processes were carried out (Type 1 through Type 4)[28]. Flax fiber mat, Biotex Flax 2×2 twill fabric mat with an areal density of 400 g/m$^2$ obtained from Composites Evolution, Chesterfield, UK (Type 5) was also used in this study. Different types of flax fibers used in this study are described in Table 1:

Table 1. Types of flax fiber used in this study.

| Fiber Type | Description |
|---|---|
| Type 1 – Linen Flax | No further mechanical process was performed on fiber |
| Type 2 – Linen Flax | Fiber was combed ten times by “opener” machine in a rough manner |
| Type 3– Linen Flax | A 50/50 blend of optimally retted fiber and over retted fiber |
| Type 4 – Linen Flax | Fiber was passed through a pair of small fluted rollers ten times to remove remaining shive |
| Type 5 – Flax Fabric | Biotex flax fabric |

Two types of resins were used as the matrix to manufacture composite plates. The bio-based resin used in this study was produced at the Department of Coatings and Polymeric Materials of

North Dakota State University. Methacrylated Epoxidized Sucrose Soyate (MESS) resin was made by the reaction of Epoxidized Sucrose Soyate (ESS) and Methacrylic Acid. ESS was synthesized from fully esterified sucrose soyate as reported previously in [29, 30]. The MESS resin was too viscous to be used for thermoset formulations. Therefore, styrene was introduced as a reactive diluent to reduce the viscosity, as well as a co-monomer to increase the rigidity of the resulting thermoset. The resulting resin contained 30% styrene. The resin was mixed with tert-butyl peroxybenzoate 98% (Luperox® P) as a high temperature initiator, cumyl hydroperoxide 45% (Trigonox 239A) as a room temperature initiator, and cobalt naphthenate (CoNap) as a promoter. The mixing ratio of Luperox® P, Trigonox 239A and CoNap were 2, 3, and 1 wt%, respectively. Styrene, Luperox® P and cobalt naphthenate were purchased from Sigma-Aldrich Co. located in St. Louis, Missouri, USA. Cumyl Peroxide commercially available as Trigonox 239A, was generously provided by AkzoNobel Co. Located in Amsterdam, Netherlands. The second resin used in this study was a vinyl ester (VE) system Hydropel® R037-YDF-40, generously provided by AOC resins Co. located in Collierville, Tennessee, USA. The hardener for VE resin was a 2-butanone peroxide (Luperox® DDM-9) solution, which was obtained from Sigma-Aldrich Co. St. Louis, Missouri, USA. Mixing ration of VE and hardener was 100 to 1 weight parts.

### 2.2 Manufacturing composite plates

The same method as described in previous work by the authors[31] was used to manufacture composite samples. Composite panels were manufactured using a hand-layup compression molding process. As mentioned, for each plate 50±4 g of fiber was placed in the mold and 250 g of resin was poured onto the fiber until the fiber was fully soaked in resin. A nonporous PTFE sheet was placed on top of the fiber and a caul plate with dimensions of 200 mm by 150 mm was placed on top of the fiber layup. The entire layup was sealed under a layer of vacuum bagging and five metric tons of force was applied using a shop press. The applied force resulted in 1.6 MPa of pressure over the composite. A schematic of the composite plate manufacturing layup is shown inFigure 2. All of the composite plates using VE resin were under pressure for 24 hours at room temperature and then post cured at 80 ºC for 12 hours. The panels using MESS resin were under pressure for 12 hours at 23.8 ºC, one hour at 150 ºC, one hour at 175 ºC and two hours at 200 ºC. To avoid warpage, the panels were cooled down to room temperature under pressure. The resulting composite plates had average thickness of 3 mm. All samples were kept in the oven at 80 ºC prior to testing.

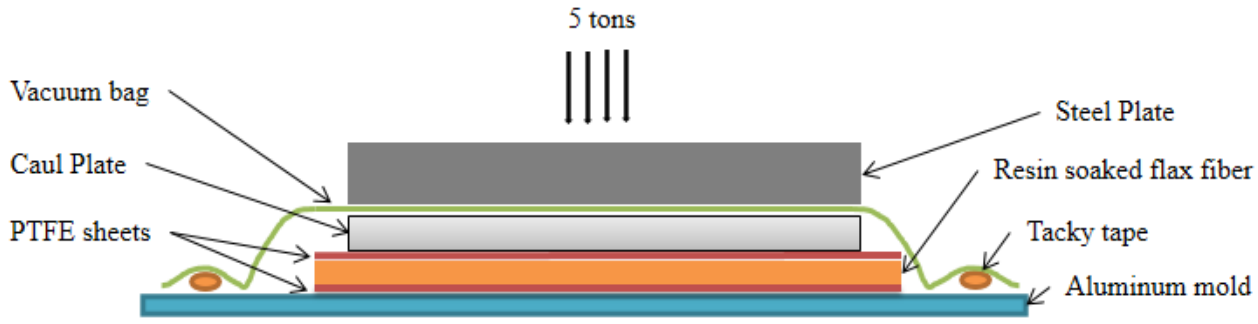


Figure 2. Composite panel manufacturing layup.

### 2.3 Void fraction measurement using conventional method

Density testing of flax fiber composites was conducted using a Mettler Toledo 33360 density determination kit. In this method composite specimen is weighed (dry), and then submerged in a measuring fluid. For this study, canola oil was used as submerging liquid. The liquid and submerged specimen were placed in the vacuum chamber under pressure of -90 kPa for 10 minutes and then the submerged fiber was weighed again. The density of composite is calculated by the equation:

$$\rho = \frac{w_o}{w_o - w}\rho_o \tag{5}$$

where, $w_o$ is the weight of the composite sample, $w$ is the weight of composite measured when submerged in the fluid, and $\rho_0$ is the density of the fluid which was determined using standard density specimen.The density measurement kit used in this study is shown in Figure 3.

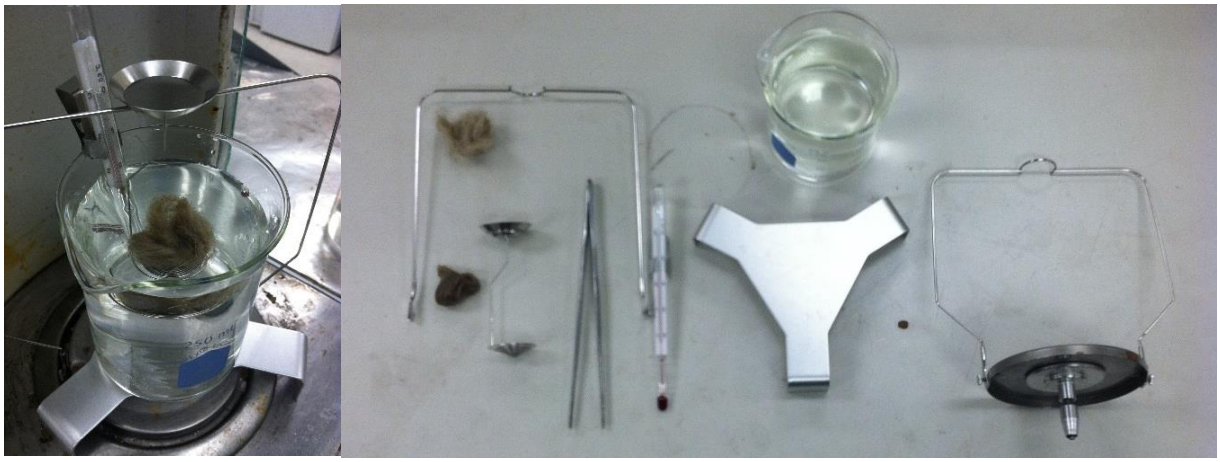

Figure 3. Mettler Toledo 33360 density determination kit.

### 2.4 Void fraction measurement using 3D images

In order to acuire 3D scans of the composite samples, small specimens were cut out of manufactured samples using a diamond blade saw. The specimens were attached to a glass rod using hot glue and placed into a GE Phoenix v|tome| x s X-ray computed tomography system (MicroCT) (Wunstorf, Germany) equipped with a 180 kV high power nano-focus X-ray tube xs|180nf and a high contrast GE DXR250RT flat panel detector. Nine hundred projections of each sample were acquired at a voltage of 100 kV and a current of 150 mA using a molybdenum target. Detector timing was 1000 ms and the total acquisition time was 1 hour and 6 minutes. Sample magnification varied per sample and approximately 20x producing a voxel size range of around10-12 microns. The acquired images were reconstructed into a volume data set using GE datos|x 3D computer tomography software version 2.2 (Wunstorf, Germany). The reconstructed volume was then viewed and processed using VGStudio Max by Volume Graphics (Charlotte, NC).

### 2.5 MATLAB® image processing code

A MATLAB® code was developed to read, analyze, and calculate the void volume in each sample. The developed code was written as follows:

```
% Start

Avg=0; TotalWT=0; Total=0;
% set the directory where the images are located
srcFiles = dir('Z:\Research\Conference papers\2016- Sampe\image\*.tif');

for i = 1 : length(srcFiles)

filename = strcat('Z:\Research\Conference papers\2016-
Sampe\image\',srcFiles(i).name);
    I = imread(filename);                                    % read the image
    I2 = imcrop(I,[65.5 15.51 191.98 392.98]);               % crop the image
    BW = im2bw(I2,0.140);                          % convert to black and white

    TotalWT=nnz(BW)+TotalWT;                  % find number of white elements
   Total=numel(BW)+Total;                    % find number of total elements

end
VoidPercentage=(1-(TotalWT/Total))*100 % find the percentage of black area

% End
```

## 3. RESULTS

### 3.1 Results from conventional method

Results of fiber volume fraction and void fraction measurements using density measurements and Equations 1-4 are presented in Table 2.

Table 2. Results of fiber volume fraction and void fraction in manufactured composite samples.

| Composite description | Fiber volume fraction | Void fraction |
|---|---|---|
| Type 1 – VE | 34.50% | 9.14% |
| Type 2 – VE | 39.13% | 8.24% |
| Type 3 – VE | 37.75% | 8.29% |
| Type 4 – VE | 36.60% | 7.68% |
| Type 5 – VE | 30.13% | 6.88% |
| Type 1 – MESS | 31.43% | 13.46% |
| Type 2 – MESS | 33.45% | 13.54% |
| Type 3 – MESS | 29.80% | 13.43% |
| Type 4 – MESS | 34.47% | 11.58% |
| Type 5 – MESS | 23.60% | 7.23% |

A typical 3D micro-CT image is shown in Figure 4. As observed, micro-CT can reveal any manufacturing flaws in the specimen, such as voids, delaminated plies and cracks.

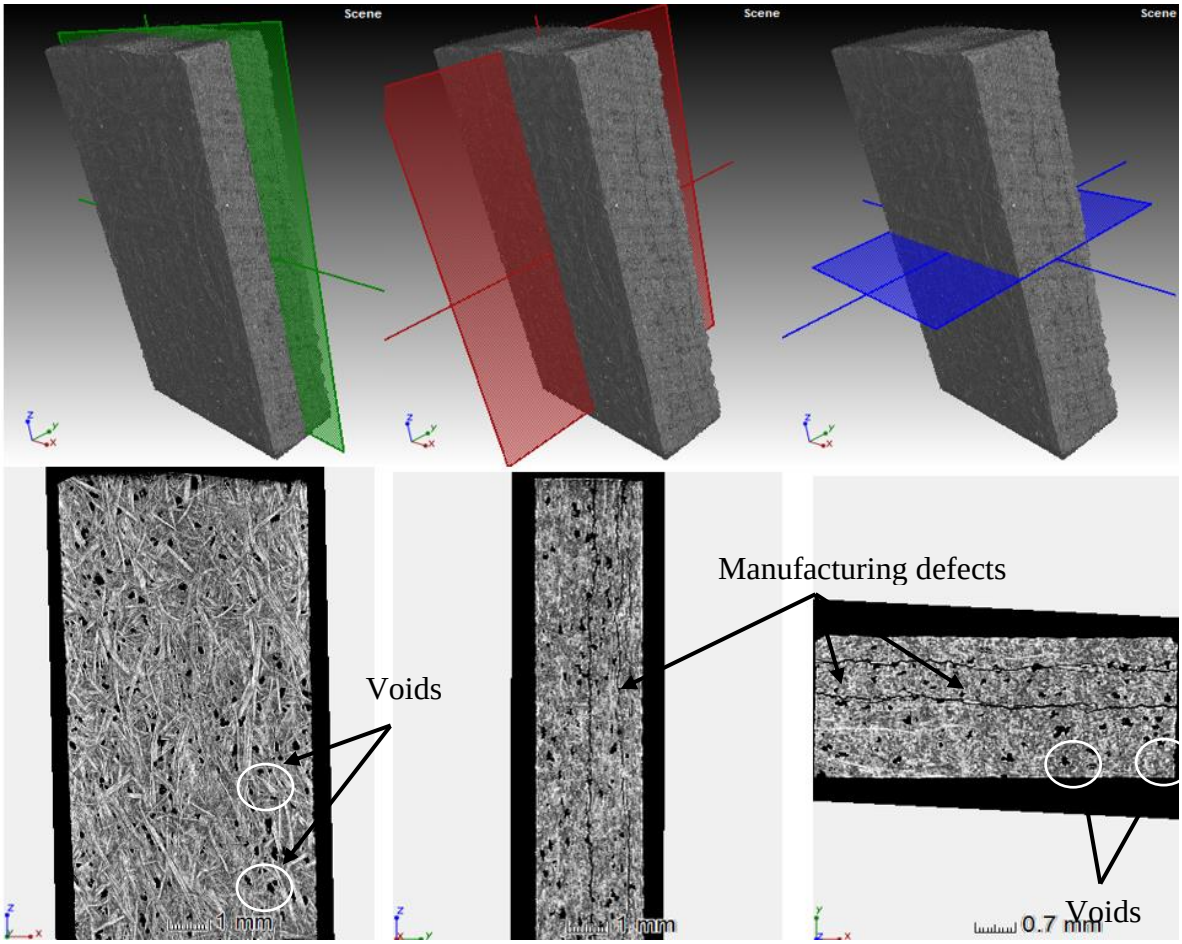


Figure 4. A typical micro-CT scan of a specimen from a composite panels.

In order to analyze the images and find the percentage of voids, after loading each image into the MATLAB® code, the opacity of the image is changed so as there is only black and white areas in the image. The white areas in the image are indication of fiber and matrix while black areas are either cracks or voids present in the material. In addition, the image is trimmed to remove extra black area in the margin of the image that may cause errors in the results. The original image and modified image is shown in Figure 5. After these corrections are completed, the percentage of black area is calculated for one image, and the average over all images (300 images for a 3mm thick specimen) processed from one specimen is calculated.

Table 3 compares the results of void fraction measurements by conventional method and use of the image processing method described. In addition, the percentage differences in the results are presented. As observed, the void content measured by micro-CT was consistently lower than that determined from immersion density. This is due to the fact that density measurements has some level of error and even after placing the specimen submerged in the liquid under vacuum pressure, there are still some micro-bubbles attached to specimens. These bubbles will count

towards the amount of voids present in the material (the measured density is lower than actual density).

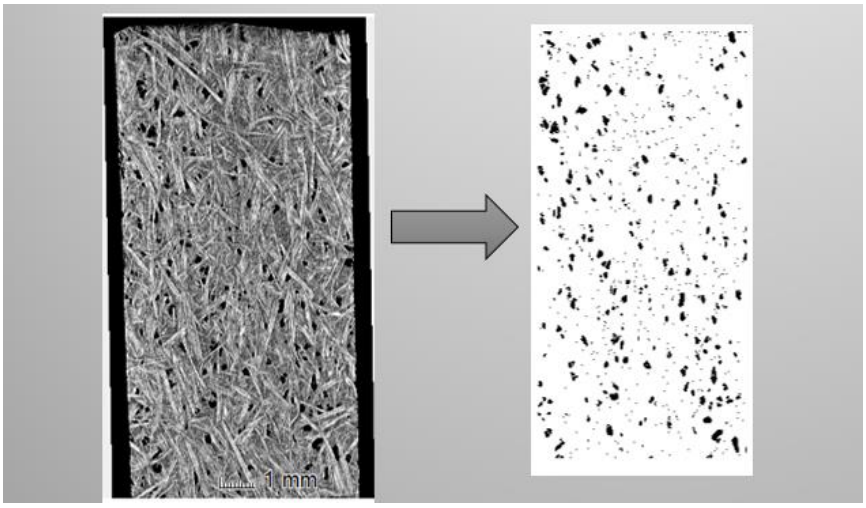


Figure 5. Correction on images done by the MATLAB® code.

As mentioned earlier, using submersion method to find the density of natural fibers and composites is a challenging task and never free of errors. Errors can be introduced at any stage of the measurements. One alternative is to find the density of natural fibers with more accurate methods such as gas pycnometry. One disadvantage of this method is that it is not available in every lab setup. On the other hand, using micro-CT can be accurate and an easier method to substitute for conventional methods, but this method also might not be available to everybody in all lab setups.

Table 3. Comparing results of void fraction measurements.

| Composite description | Void fraction | | |
|---|---|---|---|
| | Conventional method | Micro-CT scans | Difference (%) |
| Type 1 – VE | 9.14% | 9.03% | 1.2 |
| Type 2 – VE | 8.24% | 7.98% | 3.15 |
| Type 3 – VE | 8.29% | 7.94% | 4.22 |
| Type 4 – VE | 7.68% | 7.21% | 6.12 |
| Type 5 – VE | 6.88% | 6.40% | 6.97 |
| Type 1 – MESS | 13.46% | 13.23% | 1.71 |
| Type 2 – MESS | 13.54% | 13.32% | 1.62 |
| Type 3 – MESS | 13.43% | 13.22% | 1.56 |
| Type 4 – MESS | 11.58% | 11.36% | 1.89 |
| Type 5 – MESS | 7.23% | 7.12% | 1.52 |

Void fractions of ten flax fiber reinforced composite samples were calculated using two different methods; density measurements using sets of theoretical equations, and using 3D scans of composite samples and image processing with MATLAB® code. There were between 1.2% and 6.97% difference between the results obtained from the two methods. The focus of this study was to prove that using micro-CT scans is a feasible way of evaluating the quality of composites structures and with this novel method finding void fraction of manufactured composites is possible. Further investigation is required to confirm the accuracy of this method.

## 4. ACKNOWLEDGEMENTS

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