

## The Effect of Filler Size on the Presence of Voids within Resin Composite

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### Abstract

To assess the effect of filler on the presence of voids within resin-composite.

Eight Light cure resin-composites were used in this study, seven models and one commercial composite. A disc (6mm in diameter and 2mm thickness) was used to prepare samples (n=8). Each sample was then scanned, reconstructed and analysed using Micro-CT.

The percentages of voids obtained were then imported into SPSS and analysed using ANOVA and post hoc methods to check any significant differences between materials tested ( $p < 0.05$ ).

The Mean (SD) of % of voids for each group were obtained which ranged from 0.28% within Tetric Ceram to 3.48% within I4 model composite (1500nm). The different between means was statistically significant at  $p < 0.05$ .

The filler size and distribution has an effect on the percentage of voids in resin composite which was greater with larger filler content.

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### Introduction

Resin composite is the most widely used tooth-colored restorative material. It comprises of: organic resin matrix, inorganic filler, coupling agent, coloring pigments, and polymerization initiator and inhibitor. The filler component of resin composite is added to resins in order to improve their properties<sup>1</sup> and it is still the key for improving the mechanical properties including flexural strength, modulus of elasticity,<sup>2</sup> compressive strength and hardness,<sup>3</sup> as long as it is well bound to the resin matrix.<sup>4</sup> Also, the increase of filler content has an influence in minimizing polymerization shrinkage. There is a limit on the maximum possible fraction of filler that can be incorporated into a resin. As the filler fraction increases, so does the packability and the viscosity of the material,<sup>5</sup> a weight fraction of

above 80% results in a material that is stiff and not easy to manipulate.<sup>6</sup>

These filler particles have been altered in terms of their size to improve the material properties, and most of resin composites are now incorporating nanofillers ranging from 5nm to 100nm. The nanofillers aim to improve surface smoothness and gloss, polymerization shrinkage and biocompatibility without altering mechanical properties.<sup>7-9</sup>

The effect of filler particle volume fraction has been extensively studied particularly with respect to mechanical properties,<sup>2</sup> but also shrinkage. Filler particles size has attracted less attention with some reports characterizing its effect on mechanical properties,<sup>10</sup> shrinkage,<sup>11</sup> and on the elastic properties of resin composite material.<sup>5</sup> Other, has assessed the correlation between fillers, fracture toughness and presence of voids.<sup>12</sup>

Given the problems outlined above, most commercial composites currently available, do not contain one type of filler, but have a combination of sizes, i.e. they are multimodal. This aims to achieve maximum filler load with superior mechanical properties whilst maintaining

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good surface finish. Typically, two or three sizes of filler are employed (i.e. bimodal or trimodal blends), with the 'spaces' between large filler particles being occupied by filler of smaller size: these are classed as hybrid composites.

Voids within resin composite restorations and their negative effect has been previously investigated,<sup>13,14</sup> these voids are present in the restoration either due to manufacturing process or during handling and packing technique. The relation of the viscosity of resin composites (flowable-composites) and voids has also been studied and found that flowable-composites reduce voids within a class II restoration.<sup>15</sup> There is no doubt that the improved filler technology and their size has enhanced the properties of resin composite material, however the relation of filler size and the voids within the material is still not understood.

The aim of this study was to study the effect of filler size on the percentage of voids with resin composite in 3D using a micro computed tomography (Micro-CT).

### Materials and methods

The resin composites used in this study were all visible-light-cured [VLC] and comprised of 7 model composites (Ivoclar Vivadent, Schaan, Liechtenstein) and one commercial composite (Tetric Ceram, Ivoclar Vivadent, Schaan, Liechtenstein). All resin composites used were composed of the same matrix which was a combination of Bis-GMA, UDMA and TEGDMA. All of the model composites had a particulate dispersed phase of the same volume fraction (56.7%), which was treated with a silane coupling agent. The filler particles were either irregular particles of ground glass melts or silica spherical particles. The composition of resin composites is illustrated in Table 1.

A Teflon mould was used to prepare eight disc samples (6mm in diameter and 2mm thickness) of each material. Each was cured for 20 s from each side to ensure curing depth using a halogen light curing unit (Optilux 501, Kerr SDS, Peterborough, UK) with an irradiance of 550 mW/cm<sup>2</sup> and then smoothed using silicon carbide water proof abrasive paper (150 CW) and then (600 CW). Samples were placed into the chamber of a micro-CT (SkyScan1072, SkyScan, Kontich, Belgium) with a fixed current of 98mA and voltage of 104kv. Pilot images were taken to allow adjustment of scanning

parameters for good quality scanned images which were similar for all samples. Parameters used with micro-CT are summarized in Table 2.

Resin composite	Filler Particles (Ground Glass [Ba-Al-B-silicate glass])				Matrix
	Shape	Size (nm)	Wt%	Vol%	
I1	Irregular	450	76.4	56.7	BisGMA, UDMA, TEGDMA
I2	Irregular	700	76.4	56.7	
I3	Irregular	1000	76.4	56.7	
I4	Irregular	1500	76.4	56.7	
I5	Irregular	450, 1000 (1:3)	76.4	56.7	
I6	Irregular	450, 700 & 1500 (1:1:3)	76.4	56.7	
SP	Spherical	100	72.4	56.7	
TC Lot F53738	Irregular & Spherical	40, 200 & 1000	79	60	

**Table 1.** Compositions of resin composite materials used in the study.

Parameters	
Magnification	24.3
Pixel	11.24µm
Y-position	6mm
Rotation	180°
Rotation Step	45°
Exposure time	4 Sec
Gain	1
Averaging	1 frames
Filter	No
Scanning time	32 minutes

**Table 2.** Parameters used with Micro-CT in the study.

Each sample was then scanned and resultant coronal and sagittal views were then stored in 16 bit TIFF files (using N-recon software, SkyScan, Kontich, Belgium). Images for each sample were then converted into a 3D image (using CTAn software, SkyScan, Kontich, Belgium), in which binary images were obtained. A 3D model was created using CTvol software (SkyScan, Kontich, Belgium). The percentage of voids within each sample was calculated. Data were imported into

statistical software package (SPSS ver 20.0, USA) and analysed using One-Way ANOVA ( $p < 0.05$ ). Prior to post-hoc tests, data were analysed for equal variances using Levene's test for homogeneity ( $p < 0.05$ ), and as equal variances could not be assumed Dunnett's T3 was applied.

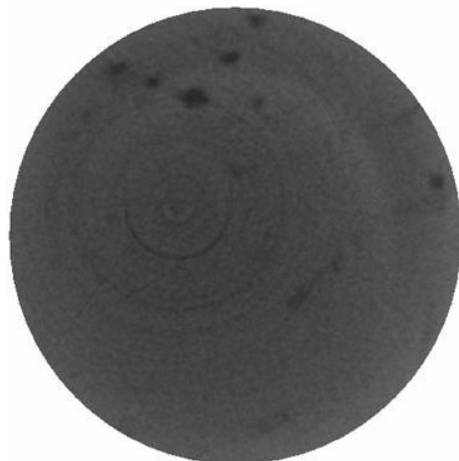
### Results

Voids were seen in the 2-D reconstructed images (Figure 1) as well as in the 3-D model (Figure 2a and 2b) constructed and viewed by the aid of CTVol software. The voids % varied from 0.28 % for TC (40:200:1000nm) to 3.48% for I4 composite (1500nm). Sp (100nm) exhibited the lowest % voids (0.44%) amongst the unimodal composites.

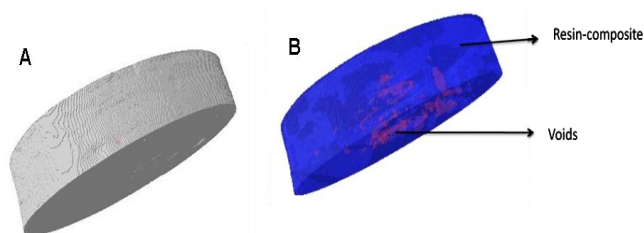
Generally, the percentage of voids increased with increasing filler size ( $r = 0.97$ ) (Figure 3). The difference of the %voids between the materials was statistically significant ( $p < 0.05$ ). The mean and standard deviation (SD) of all materials tested is summarized in Table 3.

Material	Voids%
Sp	0.44 (0.03) <sup>a</sup>
I1	0.57 (0.03) <sup>b</sup>
I2	1.47 (0.16) <sup>c</sup>
I3	3.0 (0.04) <sup>d</sup>
I4	3.48 (0.05) <sup>d</sup>
I5	0.69 (0.02) <sup>e</sup>
I6	0.36 (0.03) <sup>f</sup>
TC	0.28 (0.05) <sup>g</sup>

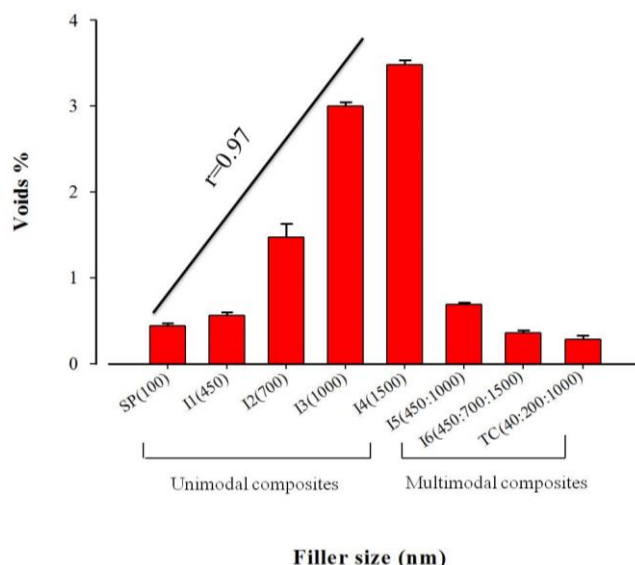
**Table 3.** Mean (SD) values of Voids % of all resin composites tested. Different small letter superscript indicates significant difference between the groups ( $p < 0.05$ ).



**Figure 1.** 2D reconstructed image of I3 sample.



**Figure 2.** 3D model of resin composite sample (A) and (B) 3D image with pseudo color (red represents the voids and blue represents resin composite).



**Figure 3.** Bar Char of Mean (SD) of all materials with Correlation between Filler Size of unimodal composite and % of Voids.

### Discussion

The use of model resin composites with controlled formulations to evaluate a property (e.g. voids percentage), allows for more meaningful comparisons to be drawn. The model resin composites used in this study had irregular or spherical shaped filler particles in nanometre range and filler volume fraction was  $< 60\%$  thus can be classified as a densified midway filled resin composite<sup>16</sup>. As it is evident from Table 5.3, the percentage of voids within multimodal TC material (40:200:1000nm) was the lowest with mean value of (0.28%), followed by unimodal Sp material (100nm) with mean value (0.44%). This low percentage of voids could be due to agglomeration and formation of large aggregates of smaller fillers.<sup>17</sup> These aggregates of smaller fillers as in TC material has proven in previous study to have the lowest water sorption valued

among other tested materials.<sup>18</sup> From these results, it is clear that both size and distribution have an influence in the presence of voids within resin composites; which is in harmony with previous study.<sup>12</sup>

The highest percentage of voids was recorded within I4 unimodal model composite (1500nm) with mean value of (3.48%). These figures illustrated the effect of filler size and distribution on the presence of voids. The correlation between the percentage of voids and filler size was a strong correlation ( $r=0.97$ ), thus the second null hypothesis was rejected.

These voids can arise during manufacturing processes or during handling and mixing procedures.<sup>19</sup> Voids may cause drawbacks when they are present within a restoration depending on where they are located<sup>20</sup> such as: marginal leakage and discolouration when present at the margins, increased wear due to the stress concentration around voids, decreased flexural strength if located between the layers of the restoration, may be misdiagnosed as secondary caries in the radiograph and also can cause incomplete adhesion between the resin composite and dentine.<sup>21</sup>

Despite the negative aspects that voids may cause when present within the resin restoration, it has been suggested that their presence may have a potential benefit as they may decrease shrinkage stress development due to the inhibiting effect of oxygen within the voids during the setting reaction and also as increased free surface within the restoration.<sup>22</sup>

Voids within resin composite have been measured in previous studies by different methods,<sup>14,18,23-26</sup> all of which were destructive and use low magnification tools. In the current study the use of micro-CT allowing measurement of voids in 3-D and enhanced characterization in a non-destructive manner, and has previously been used to correlated voids to fracture toughness of resin composite material.<sup>12</sup>

## Conclusions

With the limitation of this study it was concluded the percentage of voids was i) significantly higher with larger filler compared to smaller filler size, and ii) higher with composites of unimodal distribution than tri-modal distribution.

## Disclosure statement

The authors declare no conflict of interest.

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