



**Tufts**  
UNIVERSITY

School of  
Dental Medicine

**The Influence of Resin Cement on Marginal Microleakage  
and Fracture Strength of CAD/CAM Generated Lithium  
Disilicate Crowns**

A Thesis

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by

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## ABSTRACT

### *Aim and hypothesis*

This study aimed to evaluate fracture strength and microleakage of CAD/CAM fabricated lithium disilicate crowns cemented with two resin cements. It was hypothesized that fracture strength of lithium disilicate crowns cemented with RelyX Ultimate is higher than those cemented with Variolink II. Additionally, it was hypothesized that marginal microleakage of lithium disilicate crowns cemented with RelyX Ultimate is higher than those cemented with Variolink II.

### *Materials and method*

Fifty four extracted human molars were prepared in a standardized manner for full crown coverage. The samples were scanned and designed using E4D system. The crowns were milled out of IPS e.max CAD blocks using E4D milling center. The samples were randomly divided into four groups; 1A, 1B, 2A and 2B. For both the 1A and 1B groups, the crowns were cemented with Variolink II. For the 2A and 2B groups, the crowns were cemented with RelyX Ultimate. All groups were subjected to 5,000 thermocycles. Then, groups 1A and 2A were loaded until catastrophic fracture occurred. Groups 1B and 2B were immersed in silver nitrate dye then sectioned buccolingually and the microleakage were measured under a stereomicroscope at 1.25 magnification.

### *Results*

For fracture strength, the mean for group 1A was 2434.6 N (SD = 531.8), and for group 2A, the mean was 2453.1 N (SD = 471 N). The difference was not statistically significant (p-value = 0.937). For microleakage, the median for group 1B was 11.4% (IQR = 6.3%) and for group 2B, the median was 9% (IQR = 8.7%). The difference was not statistically significant (p-value = 0.438).

### ***Conclusion***

There was no evidence that the choice between RelyX Ultimate and Variolink II influences the microleakage and fracture strength of CAD/CAM fabricated lithium disilicate crowns. In addition, the lithium disilicate crowns' fracture strength using both cements was clinically acceptable.

## **DEDICATION**

To my wife, Fatimah, for your endless support and sacrifice.

To my mother, Zahra, for your boundless guidance and encouragement.

To my father, Noah. You are always in my heart.

I dedicate every beautiful thing in my life to all of you.

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## **LIST OF ABBREVIATIONS**

CAD/CAM: Computer-aided design and computer-aided manufacturing.

Bis-GMA: Bisphenol A-glycidyl methacrylate.

HEMA: Hydroxyethylmethacrylate.

MDP: 10-Methacryloyloxydecyl Dihydrogen Phosphate.

## LIST OF SYMBOLS

N: Newton.

$\mu\text{m}$ : Micrometer.

mm: Millimeter.

$^{\circ}\text{C}$ : Celsius.

h: Hour.

wt%: Weight percent.

SD: Standard deviation.

IQR: Interquartile range.

**The Influence of Resin Cement on Marginal Microleakage and Fracture Strength of CAD/CAM Generated Lithium Disilicate Crowns**

## Introduction

Lithium disilicate is a strong ceramic material that can be used to fabricate full anatomical crowns using CAD/CAM technology.<sup>1</sup> Similar to all-ceramic restorations, their mechanical properties, adequate cementation and bonding, accurate adaptation and reasonable esthetics determine the success of these crowns.<sup>2</sup> It has been reported that two of the main causes of all-ceramic restorations failures are catastrophic fractures and secondary caries.<sup>3</sup>

One of the critical parts of the indirect restorative process is dental luting cement. Its thickness, chemical composition and elastic modulus play an important part on the longevity of restoration.<sup>4-6</sup> For example, low bonding quality of luting cement has a negative influence on the load-bearing capacity of ceramic restoration.<sup>7</sup> Another example, some dental cement, such as resin cement,<sup>8</sup> has an ability to absorb fluid which increases the possibility of microleakage.<sup>9</sup>

For all-ceramic crowns, resin cement is recommended due to its ability to bond to tooth structure as well as silica-based ceramic.<sup>10,11</sup> However, its tendency to absorb water and undergo hydrolytic degradation can affect adversely both fracture strength and microleakage of ceramic restorations.<sup>8,9</sup> Moreover, a relatively thick luting cement layer can increase the amount of water absorption<sup>12</sup> and worsen the influence on ceramic restorations.<sup>4,13</sup>

Reich et al.<sup>14</sup> showed that lithium disilicate posterior crowns fabricated by a CAD/CAM system had an axio-occlusal internal gap with a lower quartile of 173  $\mu\text{m}$  and an upper quartile of 273  $\mu\text{m}$ , and occlusal internal gap with a lower quartile of 229  $\mu\text{m}$  and an upper quartile of 319  $\mu\text{m}$ . It was claimed that the clinically acceptable internal gap

measurement can be up to 300  $\mu\text{m}$ .<sup>15-18</sup> However, a value of 50  $\mu\text{m}$  to 100  $\mu\text{m}$  is reported to ensure best resin cement performance<sup>16,17,19</sup> and 90  $\mu\text{m}$  to reduce stresses within all-ceramic restoration.<sup>20</sup>

With the discrepancies in the internal gap associated with CAD/CAM systems, the influence of luting cements' physical properties on the fracture strength and microleakage might increase, which could be related to all-ceramics restorations failures. Therefore, this in vitro study will test the influence of two different resin cements on the microleakage and fracture strength of lithium disilicate posterior crowns fabricated with a CAD/CAM system.

### ***Historical background***

#### ***CAD/CAM systems***

CAD/CAM is the acronym for 'computer-aided design' and 'computer-aided manufacturing'.<sup>1</sup> These systems automated the fabrication process of dental prostheses.<sup>1</sup> They have three main components consisting of data acquisitions unit, designing software and production machine.<sup>1</sup>

First the preparation design and the surrounding tissues are captured and transformed into digital data.<sup>1</sup> Depending on the technology used, the preparation design can be obtained directly from the patient's mouth by an intraoral device or indirectly by scanning the cast or the impression.<sup>1</sup>

Then, the data are transferred to the designing software to be processed and presented as a 3D image of the desired restoration.<sup>1</sup> Different kinds of prostheses can be designed such as inlays, onlays, crowns, FPDs and frameworks.<sup>1</sup> Usually the data are stored in basic data

format known as standard transformation language (STL) format; however, some manufacturers use their own data format.<sup>1</sup>

Finally, the restoration is produced either by milling the restoration or by any other production technology.<sup>1</sup> There are three types of milling machines available on the market according to the number of milling axes: either three-axis, four-axis or five-axis.<sup>1</sup> All three components of the CAD/CAM system determine the quality of the restoration produced.<sup>1</sup>

CAD/CAM systems can be classified into three types according to the location of the three main components of these systems.<sup>1</sup> When the three components are located in the dental office, it is called Chairside CAD/CAM system.<sup>1</sup> In the Laboratory CAD/CAM system, the three components take place in the lab after the dentist sends a conventional impression or cast to the lab.<sup>1</sup> In either case, when data are collected and processed in the dental office or in the lab, the information is sent to a milling center, and this system is called Centralized CAD/CAM system.<sup>1</sup>

In the USA market, there are four systems available with direct intraoral scanners.<sup>21</sup> They are CEREC Bluecam System (Sirona Dental System, NY), Lava C.O.S. System (3M ESPE, MN), iTero System (Align Technology, CA), and E4D System (D4D Technologies, Richardson, TX). CEREC and E4D are the only Chairside systems currently available on the market.<sup>21</sup>

All-ceramic CAD/CAM inlays have shown a success rate of 90% after 10 years and 85% after 12 and 16 years.<sup>1</sup> Moreover, adhesively cemented all-ceramic CAD/CAM restorations exhibited a survival rate of 86.9% at the 7-year recall.<sup>22</sup> The prefabricated ceramic blocks are

expected to be more homogenous and have less internal flaws as their production and formation happens under the control of the manufacturer.<sup>23</sup>

### ***E4D Dentist® system***

This system does not require the use of powder before scanning the preparation.<sup>25</sup> An optical impression is made by red laser and micromirrors that oscillate at 20,000 cycles per second.<sup>21,25</sup> The operator has to scan the area from different angles.<sup>21</sup> This scanning time may take longer compared with other systems.

A three-dimensional image is created using computer software after the preparation is scanned.<sup>21</sup> Using the design software, the 3D model can be rotated and viewed from all directions to check the preparation, design the restoration and set up the marginal and internal gap thicknesses before sending the data to the milling machine or to a lab center.<sup>21</sup>

### ***CAM/CAM systems and internal gap***

The accuracy of chairside CAM/CAM system seems questionable. Some studies showed that CAD/CAM fabricated crowns tend to have thicker internal gap than it was set.<sup>14,26</sup> For example, Mously et al.<sup>26</sup> measured the marginal and internal gap of crowns fabricated by E4D system. They used three different spacer settings at 30, 60 and 100  $\mu\text{m}$ . They had an axial internal gap with minimum reading of 69.83  $\mu\text{m}$  and highest of 199.85  $\mu\text{m}$ . Similarly, occlusal internal gap had minimum reading of 82.63  $\mu\text{m}$  and highest of 267.45  $\mu\text{m}$ .

### ***Dental Cements***

Dental cement is a material that hardens to fill or bind adjacent objects together. When dental cement is used to attach an indirect restoration to prepared tooth structure, it is called luting

cement.<sup>27</sup> They have been classified variously in literature.<sup>11</sup> Van Noort, for example, classified them into water-based (zinc-phosphate, zinc-polycarboxylate, glass-ionomer and resin-modified glass-ionomer) and resin-based luting cements.<sup>10</sup>

### ***Resin Cement***

Dental resin restorative materials are constituted of three main components. These components are an organic resin matrix, an inorganic filler and a coupling agent, which binds the two first components together.<sup>10</sup> Resin cements are mainly based on polymethacrylates, which are further subdivided into either methyl methacrylates or aromatic dimethacrylates of the bis-GMA type.<sup>28</sup> On the market, resin cements are provided in the form of powder/liquid, encapsulated or paste/paste systems, and they are either auto-, dual-, or light-cured to form the polymer matrix.<sup>11</sup>

### ***Resin Cement Classification***

Resin cement can be classified based on their polymerization mechanisms into light-cured, chemical-cured, and dual-cured or based on their adhesive scheme into total-etch, self-etch, and self-adhesive.<sup>29</sup>

Total-etch resin cements are cements that etch both enamel and dentin by using 30-40% phosphoric acid to remove the smear layer and open dentinal tubules.<sup>29,30</sup> This type of resin cement showed the highest cement-tooth bonding strength that is close to enamel bonding strength and low microleakage.<sup>29,31</sup> However, the multiple steps required to bond the restoration to tooth structure might affect the bonding adversely because there is a higher chance of contamination.<sup>32</sup>

Self-etch resin cements are cements that need a primer to be applied over the prepared tooth before the use of the cement.<sup>29</sup> These systems are meant to be easy to use, less technique sensitive and with less clinician error due to the fewer steps of application.<sup>29,33</sup> Although their bonding to tooth surface is relatively strong, total-etch systems form in general a stronger bonding to enamel compared with self-etch systems.<sup>29,30,34</sup>

Self-adhesive resin cements are cements that are able to establish adhesion, even to metal alloy and a range of ceramics without a separate adhesive or etchant.<sup>10,30</sup> The monomer in this group contains phosphoric acid.<sup>10,29</sup> The cementation in this group requires a single step of application which became popular among operators.<sup>10,29</sup> It also showed a strong bond to dentin but a weaker bond to enamel.<sup>10,29</sup> In contrast, the use of etchant with this group weakens the dentin bond and strengthens the enamel bond.<sup>29</sup> Therefore, by selectively etching the enamel, an overall stronger bond can be achieved.<sup>29</sup> This group is not recommended with veneers due to their esthetic limitation.<sup>10</sup>

### ***Degradation of resin cement***

Some dental materials, including resin cement, undergo degradation in the oral cavity.<sup>35</sup> There are two types of this degradation: mechanical and chemical.<sup>35</sup> In resin cements, hydrolytic degradation occurs due to the cement's tendency to absorb water and swell in moist environments.<sup>8,12</sup> This degradation reduces resin cements' elastic modulus, bonding strength and stiffness, and leads to gap formation.<sup>8,36</sup>

### ***Resin Cement and Ceramic***

Among these luting agents, resin cement is considered the first choice for all-ceramic restorations for its superior mechanical properties and its ability to increase the retention and

fracture resistance of overlying ceramic materials.<sup>11,37-40</sup> In addition, resin cement can create a strong attachment between tooth structure and a ceramic restoration as it bonds directly to enamel, dentin and ceramic.<sup>10</sup> This type of bonding leads to reinforcement of both ceramic and tooth.<sup>41</sup>

### ***RelyX™ Ultimate Adhesive Resin Cement***

RelyX™ Ultimate is a dual-curing resin cement from 3M ESPE that is used in combination with Scotchbond™ Universal Adhesive.<sup>42</sup> It is used with glass ceramic indirect restorations as a total-etch or self-etch resin cement and is available in four shades.<sup>42</sup> The grain size is about 13 µm and the mixing ratio is 1 part base paste: 1 part catalyst.<sup>42</sup> The cement composition is shown in Table 1.

### ***Universal Adhesives***

Universal adhesives have higher pH values than traditional ones, which make them bond to etched enamel better than unetched enamel.<sup>43</sup> They can be applied using a total-etch, self-etch or selective-etch technique.<sup>43</sup> The selective-etch technique is done by selectively etching the enamel with phosphoric acid, rinsing it and then applying the adhesive to the enamel and dentin.<sup>43</sup>

Scotchbond™ was the first universal single-bottle adhesive on the market.<sup>43</sup> It can bond to multiple materials, such as zirconia and alumina oxide abraded metal, as it contains phosphate monomer.<sup>43</sup> In addition, it does not need a separate silane to bond to ceramic as it contains one.<sup>43</sup>

## ***Variolink®II***

Variolink®II is a resin cement from Ivoclar Vivadent® that can be used as dual-curing or light-curing cement by using the base without the catalyst.<sup>44</sup> It is also considered as a total-etch resin cement.<sup>44</sup> It is available in six base shades, two catalyst shades and three degrees of viscosity.<sup>44</sup> The main particle size is 0.7 µm.<sup>44</sup> The mixing ratio is 1 part base paste: 1 part catalyst. The cement composition is shown in Table 1.

## ***Microleakage***

Microleakage has been defined as the passage of bacteria, fluids, molecules or ions through the interstices between the sides of a cavity preparation and a restoration.<sup>45</sup>

Microleakage can reduce the durability of dental restorations as it has been linked to some problems such as recurrent caries, pulp pathology, corrosion and discoloration of restorations and dental sensitivity.<sup>46-48</sup> It is difficult to detect microleakage clinically although it might be expressed by the patient as dental sensitivity.<sup>46,47</sup>

## ***Microleakage development***

There are several factors that have been related to the development of microleakage, which can be divided into oral environmental factors, dental materials factors and clinician factors.

In the oral environment, cycles of mechanical loading and heat fluctuation happen during the day. This environment might change the physical properties of dental materials and cause elastic deformation which creates microleakage.<sup>49</sup>

Secondly, the properties of dental materials might favor or reduce the development of microleakage. Factors such as polymerization shrinkage, thermal expansion, water absorption

and material compatibility with tooth structure can create gaps between the material and the tooth structure. In one study of material compatibility, adhesive resin cement showed less microleakage compared with other types of dental cements.<sup>48</sup>

The third factor involves clinician factors. The choice of suitable dental material and proper material manipulation are critical steps in preventing formation of microleakage.<sup>49</sup> For example, Ling et al. found that microleakage in composite resin was affected by the direction and angle of light curing.<sup>50</sup>

### ***Microleakage reduction and luting cement***

There have been many attempts to reduce or eliminate microleakage; however, it seems impossible to eliminate microleakage completely.<sup>9</sup> Some of these attempts were looking at dental materials with less microleakage and optimal handling, improving tooth preparation method and design, and making a better fit of the indirect restorations to the tooth.<sup>9</sup>

Rosales-Leal et al. found that adhesives with low shear bond strength showed lower dentin wetting and higher microleakage values than other adhesives.<sup>51</sup> Another study indicated that adhesive shear bond strength of 21 MPa may show the lowest possible microleakage value.<sup>52</sup>

### ***Microleakage test***

Many methods have been used to test microleakage in vitro.<sup>49</sup> Air pressure, fluid filtration, electrochemical and radioisotope methods are examples of methods that have been used to detect and measure the extent of microleakage.<sup>49</sup> Although it is impossible to assess the samples for a long time as the samples need to be sectioned, dye penetration has been the most widely used method.<sup>49</sup> Furthermore, the investigators observed microleakage from

limited two dimensional slices while the microleakage happens in a three-dimensional pattern.<sup>49</sup> Despite these factors, this method gains its popularity for its feasibility and simplicity.<sup>49</sup>

### ***Fracture strength***

Fracture strength of a material is defined as “the stress required to break it”.<sup>10</sup> With respect to materials, all-ceramic crowns’ strength relies on type of ceramic, bond strength between veneer and core, crown thickness and the design of the restoration.<sup>53</sup> Nevertheless, there are many factors related to fracture strength including, but not limited to, crown fabrication, ceramic materials, oral environment, substrate and luting cement.<sup>54</sup> With time and under multiple stresses in the oral environment, the microcracks in ceramic material keep propagating until the restoration gets fractured.<sup>55</sup>

### ***Fracture strength of ceramic and luting cement***

Blatz et al.<sup>56</sup> reported that cementation technique influences the fracture strength of all-ceramic crowns. Another author<sup>57</sup> has concluded that luting cement type and the type of fatigue applied affected the fracture strength of three types of dental ceramics, which included lithium disilicate.

Thompson P and Rekow D<sup>4</sup> have found that as the elastic modulus of the substrate increased, the ceramic fracture strength increased. Moreover, it seems that higher bond strength of luting cements may reduce the possibility of all-ceramic restorations fracture.<sup>58</sup> On the contrary, as luting cement thickness increased, the ceramic fracture strength decreased.<sup>4,13</sup> Therefore, they recommended limiting luting cement thickness as possible.<sup>4</sup> In addition, a thicker luting cement layer results in higher water absorption, which allows hydrolytic

degradation of resin cement.<sup>12</sup> With time, this weakens all-ceramic restoration as the stresses within the restoration are redistributed and eventually increasing.<sup>8</sup> On the other hand, these stresses might be reduced by using luting cement with high Young's modulus and a thickness of 90  $\mu\text{m}$ .<sup>20</sup>

### ***E.max CAD crowns fracture strength values***

Seydler et al.<sup>59</sup> tested the influence of wall thickness on the fracture strength of lithium disilicate crowns. They used e.max CAD blocks to fabricate posterior crowns using CEREC system. The crowns were adhesively cemented to extracted teeth with Multilink resin cement. The artificially aged samples showed mean (SD) fracture strength of 369.2 (117.8) N, 889.1 (154.6) N and 980.8 (115.3) N for crowns with wall thickness of 0.5 mm, 1.0 mm and 1.5 mm respectively. In addition, Guess et al.<sup>60</sup> recorded fracture strength ranging from 1100 N to 1200 N for crowns with 1.5 mm and 2 mm axial and occlusal thickness respectively. Bakeman et al.<sup>61</sup> tested the fracture strength of lithium disilicate (IPS e.max) partial coverage restoration cemented with Variolink II. Each restoration was fabricated with either one mm of thickness or two mm. Their mean (SD) fracture strengths were 2105 (567) N and 2505 (401) N respectively.

### ***Microleakage and fracture strength using RelyX Ultimate or Variolink II***

3M ESPE claims that RelyX Ultimate showed higher shear bond strength compared with Variolink II using total-etch technique.<sup>62</sup> On the other hand, Variolink II has higher modulus of elasticity values than RelyX Ultimate.<sup>62,63</sup> Although higher values of these mechanical properties could maximize the ceramic fracture strength, the reduction in fracture resistance

of all-ceramic crowns after aging contributes significantly more to the loss of bond strength than to the reduction of cement stiffness.<sup>8</sup>

Thereby and according to the findings in this review, using RelyX Ultimate might lower the microleakage and increase fracture strength of all-ceramic crowns.

### ***In vitro simulation of oral environment (aging)***

As it is difficult to understand all factors involved in the degradation process of dental materials, many in vitro methods were developed in an attempt to simulate the oral environment.<sup>64</sup> Two of these in vitro methods are thermocycling and mechanical loading.

Thermocycling mimics the temperature fluctuation happening in the oral cavity.<sup>64</sup> It is considered as an acceptable in vitro method for aging dental materials.<sup>64</sup> It was suggested that 10,000 cycles is equivalent to one year of clinical function.<sup>65</sup> On the other hand, researchers have found that the number of cycles had no effect on the level of microleakage.<sup>66</sup>

Mechanical loading, however, simulates the complex stresses happening in the oral environment, such as food chewing.<sup>64</sup> In literature, different numbers of cycles were reported. Two hundred fifty thousand mechanical cycles were used to resemble one year of chewing strokes. Another study<sup>67</sup> used 1,000,000 cycles in water estimating 15 months of clinical service based on a survey<sup>68</sup> reporting that a person chews 800,000 cycles a year. Kassem A.S. et al.<sup>69</sup> suggested that 1,000,000 cycles is equivalent to five to ten years of service.

In the case of bonding cement, despite the observation of the effect of mechanical loading on bond interface degradation, its mechanism is still unclear.<sup>64</sup> For that reason, some

investigators have combined it with thermocycling. In fact, it showed greater reduction in bond strength of total-etch cement to dentin compared with thermocycling or mechanical loading separately.<sup>70</sup> Seydler B et al.<sup>59</sup> compared the fracture load of lithium disilicate crowns having different wall thickness with each other. They combined 1,200,000 mechanical loading cycles with 10,000 thermocycles in order to simulate five years of clinical service.

However, studies use different aging protocols in terms of temperature range for thermocycling, magnitude of force for mechanical loading and number of cycles for both.<sup>64</sup> Usually, the reason for choosing these numbers is not given and seems arbitrary.<sup>64</sup> This makes it difficult to compare results with each other.<sup>64</sup>

## **Aim and Hypothesis**

### ***Objective***

The objective of this in vitro study was to evaluate fracture strength and microleakage of CAD/CAM fabricated lithium disilicate posterior crowns cemented with RelyX Ultimate and Variolink II resin cements.

### ***Variables tested***

- 1- Fracture load in Newtons.
- 2- Microleakage in percentage.

### ***Hypothesis***

- 1- Fracture strength of lithium disilicate crowns cemented with RelyX Ultimate resin cement is higher than those cemented with Variolink II resin cement.
- 2- Marginal microleakage of lithium disilicate crowns cemented with RelyX Ultimate resin cement is higher than those cemented with Variolink II resin cement.

### ***Clinical significance***

The results of this study may provide clinicians with information about the performance of CAD/CAM fabricated lithium disilicate crowns using the two tested dental cements.

## **Research Design**

### ***Power calculation for the microleakage analysis***

A power calculation was conducted using nQuery Advisor (Version 7.0). Assuming a mean (SD) dye penetration of 5.0% (3.6%) for RelyX Ultimate and 1.9% (2.0%) for Variolink II,<sup>71</sup> a sample size of n = 17 per group was adequate to obtain a Type I error rate of 5% and a power of 80%.

### ***Power calculation for the fracture strength analysis***

A power calculation was conducted using nQuery Advisor (Version 7.0). Assuming a mean (SD) fracture strength of 2026.8 (364.5) N for RelyX Ultimate<sup>72</sup> and 980.8 (115.3) N for Variolink II,<sup>59</sup> a sample size of n = 10 per group was adequate to obtain a Type I error rate of 5% and a power greater than 99%.

### ***Inclusion criteria***

- Extracted human molars.

### ***Exclusion criteria***

- Teeth with caries (including root caries).
- Teeth with restorations.
- Teeth that were root canal treated.
- Teeth with cracks that extended to the cemento-enamel junction.
- Teeth with cervical defects (e.g., erosion or abrasion).

## **Materials and Methods**

Fifty four freshly extracted intact human molar teeth were collected and placed in labeled jars containing a liquid sterilant (0.5% sodium hypochlorite, Fisher Chemical, NJ); teeth were collected from the common jar in the oral surgery department at Tufts University School of Dental Medicine.<sup>73</sup> The teeth were collected in a de-identified manner and are not traceable to the patient they were obtained from.

The selected teeth were free from caries and restorations. They were cleaned of surface debris and stains with a hand scaler, and were stored in tap water at room temperature throughout the course of the study to prevent teeth desiccation.

The roots of all selected teeth were notched for retention and embedded along their vertical alignment with the cemento-enamel junction positioned 1 mm above the top of the mounting template. The template was filled with auto-polymerizing acrylic resin (Caulk Orthodontic Resin, Dentsply Caulk, Milford, DE) to secure the extracted teeth.

After mounting, the occlusal surface of each mounted tooth specimen was sectioned flat five mm above the top of the acrylic resin with a thin slow-speed sectioning saw (11-4254-blade, Isomet; Buhler Ltd, Evanston, IL).

A high-speed handpiece (Midwest Dentsply, Des Plaines, IL) was secured in a surveyor (Degussa F1; DeguDent, Hanau, Germany) so that a coarse diamond tapered rotary cutting instrument (847-16; Henry Schein, Melville, NY) was oriented at a four degree angle from the vertical axis of the tooth to create a standardized total occlusal convergence angle of 14 degrees (Figures 1 and 2).

The mounted teeth were secured vertically in a custom jig made of polysiloxane (Lab-Putty, Coltene, Switzerland) which were held firmly in a surveyor base. The surveyor was adjusted to be parallel to the floor.

Axial reduction was accomplished by rotating the mounted tooth against the rotating bur. The diamond rotary cutting instrument selected is recommended to create a shoulder finish line and to provide an optimal preparation for interpretation by the E4D CAD/ CAM system.

Using water spray, the axial surface was reduced to create a shoulder with depth of 1 to 1.5 mm and axial length of approximately four mm, using a new diamond rotary cutting instrument for each tooth specimen. Then, the coarse diamond bur was replaced with a fine bur (856—16; Henry Schein, Melville, NY) fitted in the handpiece to smoothen the surface of the preparation (Figure 3).

The prepared teeth were scanned using the E4D dentist CAD/CAM system (Figure 4). This scanner uses optical imaging of the prepared tooth from different directions to achieve a high resolution image.

The finish line was set and adjusted as necessary. The crown was designed to have wall thickness of one mm apically, two mm in the contact area and 1.5 mm in the other areas. The cement space was set at 60  $\mu$ m starting 0.25 mm above the margin. Then the crowns were milled out from lithium disilicate blocks (IPS e.max CAD, Ivoclar, Buffalo, NY) using the E4D milling center (Figures 4 and 5). The milling center was calibrated and maintained 24 hours prior to the milling process. After calibrating a Programat P500 furnace (Ivoclar, vivadent) (Figure 6), all the crowns were fired using the manufacturer's recommended settings.

After preparation and crown milling, the crowns were fired using a Programat P500 furnace. Then, each sample was given a number and the surface area of the prepared tooth was calculated and recorded. The specimens were divided randomly, using block randomization on tooth size, into four groups (1A, 1B, 2A, 2B) to ensure a comparable surface area for all groups (Figure 7).

The prepared teeth were cleaned with a prophylaxis brush containing water and fluoride free flour of pumice, then rinsed and dried but left physiologically moist. Each crown was placed on the prepared tooth and marginal fit was closely evaluated by one operator. Some adjustments were made as necessary to improve the seating of the crown on the prepared teeth. This was accomplished by gently grinding the internal surface of the crown with a high-speed handpiece (NL-75S, NSK America; Brasseler, Savannah, GA) and a small round diamond rotary cutting instrument (6801; Brasseler, Savannah, GA).

The catalyst and base of RelyX Ultimate and Variolink II resin cement were mixed according to the manufacturer's instructions using Monobond Plus as primer and Excite F DSC as adhesive with Variolink II, and Scotchbond Universal Adhesive as primer and adhesive with RelyX Ultimate. Each crown was lined with cement and initially seated with strong finger pressure. The assembled teeth and crown were subjected to a total axial seating force of 50 N per specimen (Figure 8) and a light cure device to initiate polymerization for the recommended time by the manufacturer.

Excess cement was cleaned from the margins and the specimens were stored in tap water at room temperature before thermocycling (Figure 9). Then, the cemented copings were thermal

cycled between water temperatures of 5 °C and 55 °C for 5,000 cycles with a 15 second dwell time at each temperature.

After aging, the crowns for groups 1A and 2A were tested for fractural strength using a universal testing machine (Model 5566; Instron Corp, Canton, Mass) at a crosshead speed of 0.5 mm/min. The loading was along the apico-occlusal axis of the tooth until catastrophic failure occurred. The force of fracture was recorded and the predominant nature of fracture was recorded by a single operator.

For groups 1B and 2B, the crowns were immersed into a solution of 50% wt silver nitrate (pH=9.5) for 24 hours, followed by eight hours in photo-developing solution (Eastman Kodak Co, Rochester, NY) in dark room. Specimens were washed under running water for one minute after which they were embedded in epoxy resin (Epo-Kwick Resin, Buehler, IL, USA). Each tooth was sectioned buccolingually using an eight inch diameter and 0.5 mm thickness wheel (Isomet 1000, Buehler, IL, USA).

Both the mesial and distal halves of each specimen were analyzed with 1.25 magnification using a stereomicroscope (SZX16, Olympus, Pennsylvania) and scored according to the degree of dye penetration along the dentinal walls based on the following formula:

$$\frac{\text{Amount of dye penetration}}{\text{Total surface area}} \times 100$$

Then, the average of the mesial and distal halves was calculated and reported as the dye penetration for each specimen (Figure 10).

All the procedures were done in the laboratories of Tufts University School of Dental Medicine and by one operator.

## **Statistical Analysis**

The Shapiro-Wilk test was used to assess the assumption of normality for each group (1A, 1B, 2A and 2B). The difference between Variolink II and RelyX Ultimate was evaluated for each outcome separately (fracture strength and microleakage). The independent-samples t-test was used for fracture strength; the Mann-Whitney U test was used for microleakage due to non-normality of the data. Means and standard deviations were calculated for normally distributed data (fracture strength); medians and interquartile ranges were calculated for non-normally distributed data (microleakage). SPSS Version 22 was used for the analyses and the significance level was set at 0.05.

## Results

For fracture strength, one sample was excluded from the Variolink II group because the sample developed cracks after aging.

The mean fracture strength of RelyX Ultimate was 2453.1 N (SD = 471 N), while the mean of Variolink II was 2434.6 N (SD = 531.8). Figure 11 shows the boxplot of each group. Both groups were normally distributed (Variolink II p-value = 0.770, RelyX Ultimate p-value = 0.768; Shapiro-Wilk test). The difference in fracture strength between the two groups was not statistically significant (p-value = 0.937; independent-samples t-test).

For microleakage, the RelyX Ultimate group showed a lower median microleakage percentage at 9% (IQR = 8.7%) than the Variolink II at 11.4% (IQR = 6.3%). Figure 12 shows the boxplot of each group. The RelyX Ultimate group was not normally distributed (Variolink II p-value = 0.628, RelyX Ultimate p-value = 0.012; Shapiro-Wilk test). The difference in microleakage between the two groups was not statistically significant (p-value = 0.438; Mann-Whitney U test).

## Discussion

It has been reported that cementation techniques and cement properties have a strong influence on ceramic strength and their susceptibility to secondary caries.<sup>4,51,56,57</sup> These two conditions are among the main causes of the failure of all-ceramic restorations.<sup>3</sup> In addition, although it is possible to set the desired thickness of the cement by adjusting the restoration internal gap at the design stage using CAD/CAM systems, the milled restorations tend to have thicker cement spaces.<sup>14,26</sup>

It is therefore assumed that with relatively thick cement space, the influence of the cement used on the CAD/CAM fabricated lithium disilicate crowns could increase. Thus, using a type of resin cement with a higher bond strength on all-ceramic crowns could lower the associated microleakage and increase the fracture strength.<sup>51,58</sup>

As RelyX Ultimate resin cement has been reported to have higher bond strength than Variolink II,<sup>62</sup> the purpose of this study was to evaluate the fracture strength and the associated microleakage of lithium disilicate crowns cemented with these two types of cement and compare them with each other.

Both null hypotheses could not be rejected. Thus, we could not detect any difference in the crowns' fracture strength and microleakage between these two types of resin cement.

In this study, the crowns cemented with RelyX Ultimate showed (mean = 2453.1 N, SD = 471 N) similar values to those cemented with Variolink II (mean = 2434.6 N, SD = 531.8 N). Although Bakeman et al.<sup>61</sup> used lithium disilicate restoration with partial coverage and a different fabrication technique, the results were similar to those found in this study. On the other hand, the observed numbers in this study were relatively higher than those reported in

other studies.<sup>59,72</sup> The higher numbers observed in the current study could be related to the bonding adhesion that was provided by both types of cement used in this study, the use of extracted teeth instead of typodont teeth and the use of a different CAD/CAM system.

The thermal fluctuation that occurred during the thermocycling procedure could have caused an interaction between the mechanical stresses and the material flaws could have led to microcrack propagation and caused the restoration to ultimately be weaker.<sup>55,67</sup> In this study, prefabricated CAD/CAM blocks were used as they were expected to have fewer flaws because the material is more homogenous.<sup>23</sup> Also, a resin cement with higher bond strength was expected to strengthen the final restoration more than a type of cement with less strength.<sup>54</sup> Borges et al<sup>57</sup> reported that the higher bond strength of luting cement may reduce the possibility of all-ceramic restoration fracture. However, this was not observed in this study. The fracture strength of the ceramic restorations showed no significant difference between both types of cement. On the other hand, these numbers exceeded those observed in other studies as the average bite force in full dentate subjects was 720 N (244 to 1243 N).<sup>74</sup>

In this study, although the crowns cemented with RelyX Ultimate (median = 9%, IQR = 8.7%) showed lower values than those cemented with Variolink II (median = 11.4%, IQR = 6.3%), the difference was not statistically significant. These values were higher than those reported by Uludag B et al.<sup>71</sup> This finding could be related to the difference in the aging protocol and the lithium disilicate crowns' fabrication process. Crowns fabricated using the pressing technique were found to fit marginally better than the CAD/CAM fabricated crowns.<sup>26</sup>

A possible correlation has been reported between the penetration depth of the adhesive resin and its shear bond strength and microleakage.<sup>51</sup> Hence, it was assumed that crowns cemented with RelyX Ultimate would show different microleakage than those cemented with Variolink II. By using CAD/CAM fabricated crowns, the estimated thicker cement layer allows more water absorption and probably more hydrolytic degradation in both types of cement.<sup>8,14,26</sup> This could promote microleakage by increasing cement degradation and gap formations between the crown and the tooth.<sup>8</sup> In addition, the bonding degradation occurs after water aging redistributes the stresses within the ceramic leading to the lower fracture resistance of the restoration.<sup>8</sup> Although a difference was observed in the microleakage values, it was not statistically significant. This could be related to the fact that both types of cement have relatively low solubility, which could have reduced the anticipated degradation and gap formation.

Extracted human molars were used in this study as recommended<sup>73</sup> in an attempt to simulate a clinical situation with regard to the bonding characteristics, modulus of elasticity and thermal conductivity.<sup>54</sup> As there was no control over the tooth sizes, the randomization was blocked on tooth size to ensure a balanced distribution over the four groups. Since available studies on microleakage and fracture strength lack standardization in areas such as sample preparation, aging and microleakage measurements, it is very difficult to compare the studies to each other.<sup>64</sup> In this study, all the samples were prepared and stored in the same manner. The E4D milling unit was maintained and calibrated 24 h by the manufacturer's technical representative prior to the milling procedure. In addition, the measurement was used in a continuous scale (percentage). This allowed us to weight the dye penetration for each sample with more information than with scoring systems.

The samples were thermocycled 5,000 times in order to mimic the temperature fluctuations that occur in the oral cavity. Although this was reported to simulate six months of service,<sup>65</sup> a study<sup>66</sup> found that increasing the number of cycles did not affect the depth of dye penetration. Therefore, it is not clear if increasing the number of cycles would show more extreme microleakage results. Moreover, in some studies, thermocycling and mechanical loading were combined to age the samples.<sup>64</sup> That method was found to decrease bond quality more than with thermocycling or mechanical loading separately.<sup>70</sup>

In this study, the cement space was set at 60  $\mu\text{m}$  to get the lowest possible marginal and internal gap based on a similar study reported by Mously et al.<sup>26</sup> However, they also found that this setting resulted in crowns with occlusal internal gaps ranging from 82  $\mu\text{m}$  to 200  $\mu\text{m}$  with a median of 150  $\mu\text{m}$ . As cement thickness of not more than 100  $\mu\text{m}$  was observed for better performance of resin cement,<sup>20</sup> it was assumed that the influence of both types of resin cement on the crowns would be higher.

However, this study has some limitations. The process of adjusting the internal surface of crowns to ensure full crown seating could change the cement thickness among the samples. Also, microleakage was measured in two dimensions after cutting the samples in two halves. Although this method was used by other investigators, creating more cutting sections would have allowed more dye penetration measurements. In addition, although the light curing was conducted by one operator in attempt to follow the instructions of the cement manufacturer and cure the cement from the same angles, the distance and angles should have been standardized as this could have an influence on microleakage.

In the future, using other types of cement with different water absorption and solubility could provide a different outcome, as would using different ceramic materials.

## **Conclusion**

Within the limitations of this in vitro study, lithium disilicate crowns fabricated using the E4D CAD/CAM system and those cemented with RelyX Ultimate or Variolink II exhibited no significant difference in microleakage or fracture strength values. In addition, the fracture strength of the crowns exceeded the maximum bite force in the case of both cement systems.

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## **APPENDICES**

Appendix A: Tables

Appendix B: Figures

## Appendix A: Tables

| Variolink®II   | RelyX™ Ultimate   | Scotchbond™ Universal Adhesive   |
|--|---|--|
| <p><b>Monomer matrix:</b></p> <ul style="list-style-type: none"> <li>• Bis-GMA</li> <li>• Urethane</li> <li>• Dimethacrylate</li> <li>• Triethylene glycol dimethacrylate</li> </ul> <p><b>Fillers:</b></p> <ul style="list-style-type: none"> <li>• Barium glass</li> <li>• Ytterbium trifluoride</li> <li>• Ba-Al-fluorosilicate glass</li> <li>• Spheroid mixed oxide</li> </ul> <p><b>Others:</b></p> <ul style="list-style-type: none"> <li>• Catalysts</li> <li>• Stabilizers</li> <li>• Pigments</li> </ul> | <p><b>Base Paste:</b></p> <ul style="list-style-type: none"> <li>• Methacrylate monomers</li> <li>• Radiopaque, silanated fillers</li> <li>• Initiator components</li> <li>• Stabilizers</li> <li>• Rheological additives</li> </ul> <p><b>Catalyst Paste:</b></p> <ul style="list-style-type: none"> <li>• Methacrylate monomers</li> <li>• Radiopaque, alkaline (basic) fillers</li> <li>• Initiator components</li> <li>• Stabilizers</li> <li>• Rheological additives</li> <li>• Pigments</li> <li>• Fluorescence dye</li> <li>• Dark cure activator for Scotchbond Universal adhesive</li> </ul> | <ul style="list-style-type: none"> <li>• MDP Phosphate Monomer</li> <li>• Dimethacrylate resins</li> <li>• HEMA</li> <li>• Vitrebond™ Copolymer</li> <li>• Filler</li> <li>• Ethanol</li> <li>• Water</li> <li>• Initiators</li> </ul> |

*Table 1: Composition of Variolink®II, RelyX™ Ultimate, and Scotchbond™ Universal Adhesive.*

## Appendix B: Figures



*Figure 1: Secured handpiece on a surveyor and the custom putty jig.*



*Figure 2: The bur at four degrees of angulation.*



*Figure 3: a sample after preparation.*



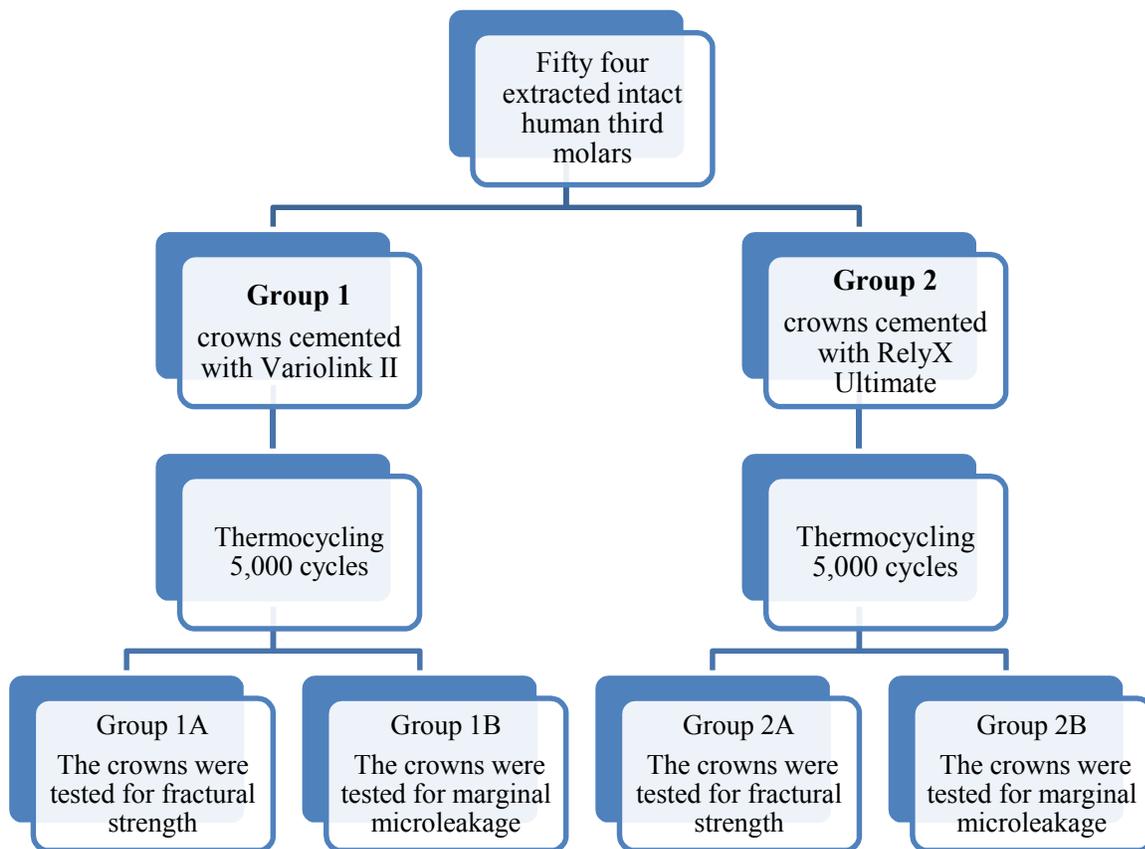
*Figure 4: E4D system. On the left, the scanning and designing unit. On the right, the milling center.*



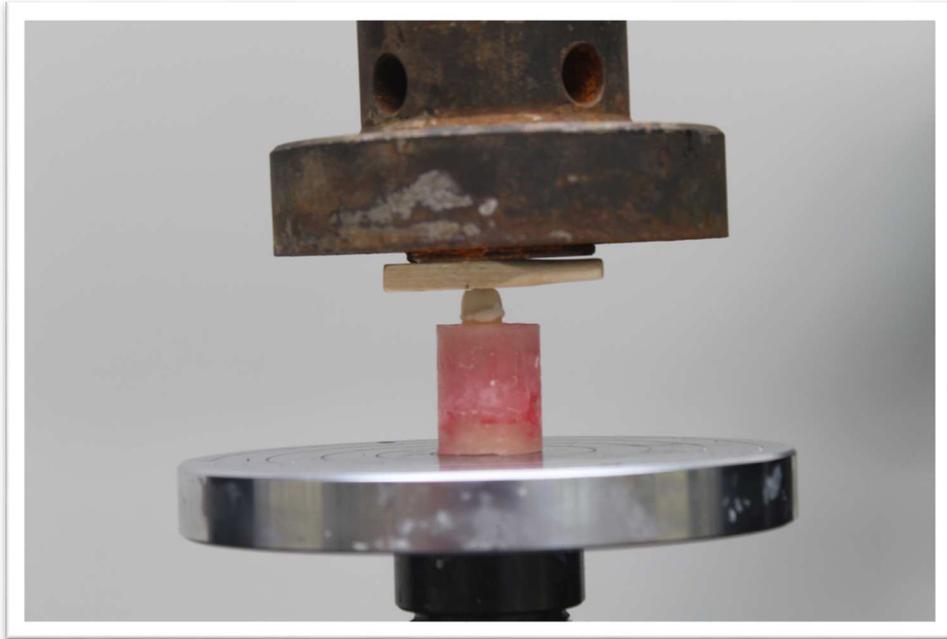
*Figure 5: Crown after milling and detaching from the block stump.*



*Figure 6: The Programat P500 furnace after calibration.*



*Figure 7: The groups and the subgroups of the study.*



*Figure 8: Seating crowns with 50 N occlusal force.*



*Figure 9: Sample number 20 after being cured and cleaned of excess cement.*

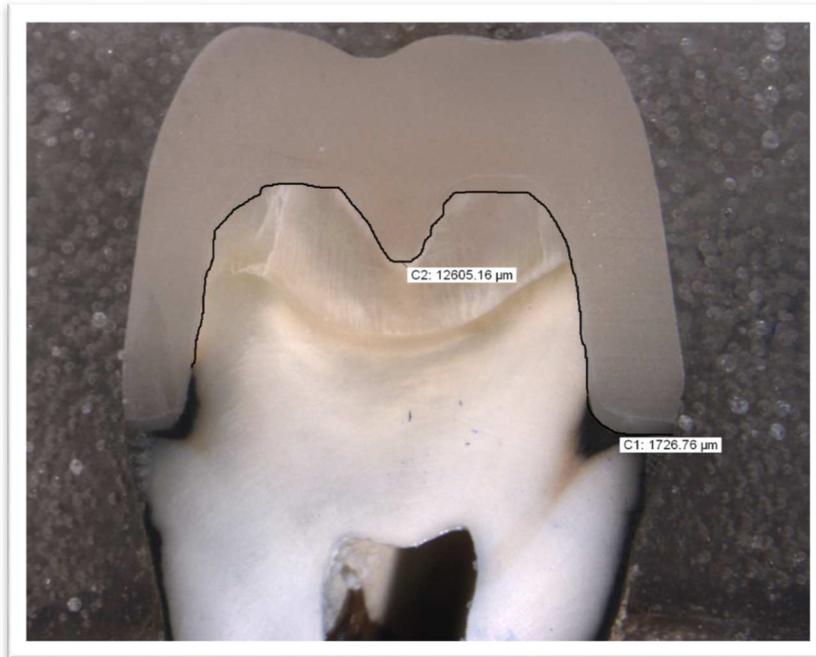


Figure 10: Tracing dye penetration and taking the measurement of the whole tooth surface.

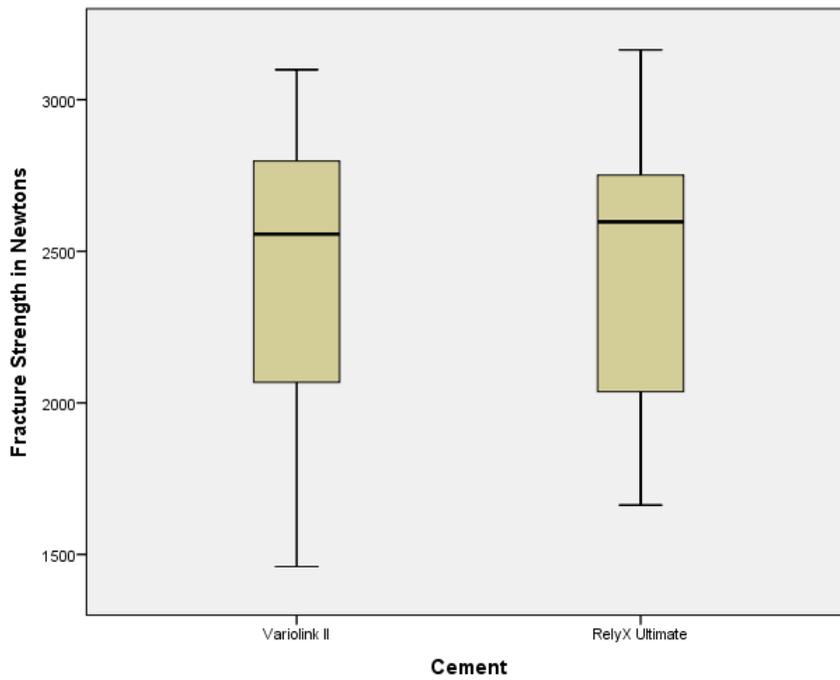
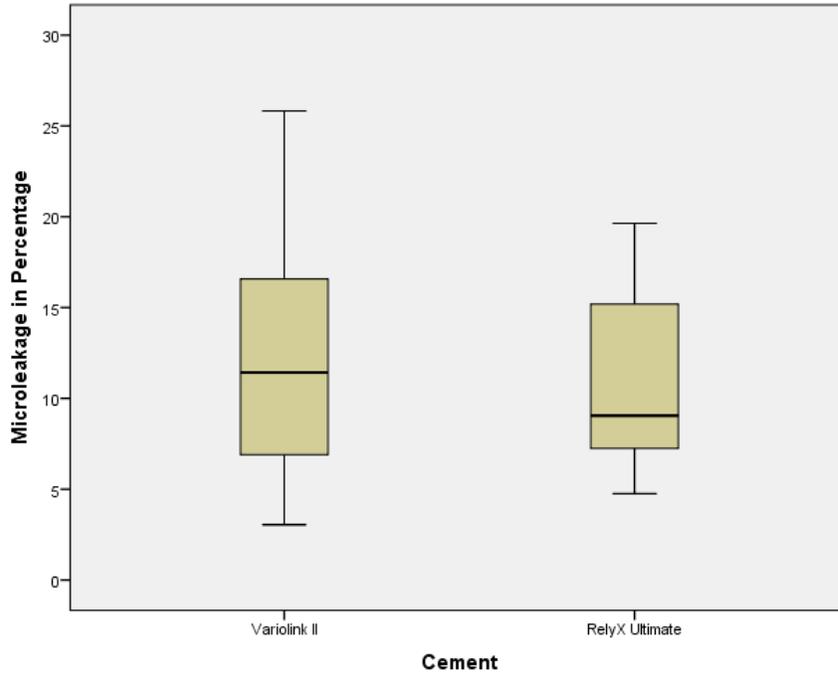


Figure 11: Fracture strength boxplots.



*Figure 12: Microleakage boxplots.*