

## EFFECT OF CEMENT FILM THICKNESS ON THE MICROSHEAR BOND STRENGTH OF CAD/CAM LITHIUM DISILICATE BONDED TO CERVICAL AND MID-CORONAL ENAMEL

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### ABSTRACT

**Objective:** The aim of this study was to compare the microshear bond strength ( $\mu$ SBS) of lithium disilicate microdiscs with cement film thicknesses of 20  $\mu$ m, 40  $\mu$ m, and 100  $\mu$ m at the coronal, middle, and cervical areas of the buccal surface of maxillary premolars.

**Methods:** 81 lithium disilicate microdiscs (IPS e.max CAD, Ivoclar Vivadent) were divided into three main groups (n=27) according to the location of enamel on the buccal surface, and then each was subdivided into three subgroups (n=9) according to cement film thickness. Group A=20  $\mu$ m, Group B=40  $\mu$ m, Group C=100  $\mu$ m. Each group was subdivided into coronal (c), middle (m), and cervical (v) areas. The buccal surfaces were prepared with a 0.5 mm depth in enamel. The cemented specimens were thermocycled for 2,500 cycles between 5 and 55°C. A universal testing machine was used for the  $\mu$ SBS test until failure. Failure modes were assessed using a stereomicroscope.

**Results:** One-way ANOVA statistical test ( $p \leq 0.05$ ) and Tukey's post hoc test showed a statistical difference in the mean  $\mu$ SBS of groups A, B, and C. Pairwise comparisons ( $p \leq 0.05$ ) showed no significant difference between groups Ac and Am, nor Bc and Bm. Significant differences were observable in all other subgroups. Failure modes showed no significant differences in their overall distribution, however, cohesive failures were predominant in groups Ac and Bm, while adhesive failures were mostly seen in Cv.

**Conclusion:** A thicker cement film thickness in the cervical area of the tooth greatly decreases  $\mu$ SBS of lithium disilicate, while microdiscs cemented coronally with a thinner film showed better adhesion.

**KEYWORDS:** cement thickness, lithium disilicate, CAD-CAM, microshear bond strength.

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## INTRODUCTION

Durability is one of the most important criteria that any dental restoration should possess. For an all-ceramic restoration to perform at its full potential, both the type of ceramic and bonding materials need to have optimal properties. Lithium disilicate has gained popularity as it combines both strength and translucency needed for an esthetic anterior restoration, namely, laminate veneers<sup>(1)</sup>. The effect of ceramic thickness on the mechanical and physical properties of laminate veneers has been widely documented<sup>(2-4)</sup>. The adhesive capacity of resin cements used for veneers has also been extensively studied<sup>(5-7)</sup>.

However, among those studies only a few have included the factor of cement thickness in this particular context<sup>(8-10)</sup>. Cement thickness is of crucial importance in the longevity of a restoration as it is the link between the ceramic-cement-tooth substrate triplex.

The facial surfaces of teeth have different enamel thicknesses, with the cervical third having the least enamel thickness and thus the greatest risk of dentin exposure during veneer preparation. Therefore, laminate veneer preparations are performed with three different depths, according to the area of the facial surface being prepared. Sound cervical enamel can often be as thin as 0.5 mm, meaning that even a minimal veneer reduction of 0.3 mm would leave only a fine layer of enamel<sup>(11)</sup>. Several studies have assessed the adhesive performance of resins on different areas of the tooth and there seems to be a consensus that the cervical region lacks in its retentive ability<sup>(12-14)</sup>.

The histology of enamel varies widely at different areas of the tooth. The culprit to this poor retentive performance would be the orientation of enamel prisms. The cervical area has disarrayed enamel prisms, as opposed to the more organized structure of the coronal and middle thirds<sup>(14)</sup>.

To date, no studies have assessed whether the thickness of a resin cement would affect the bond strength to a specific area of a tooth surface. The microshear bond strength test provides more precise measurements of bond strength as a small bonding area prevents any discrepancies in enamel preparation depth, cement thickness, and ceramic fabrication defects.

The aim of the present study was to evaluate the effect of cement film thickness on the microshear bond strength of lithium disilicate microdiscs placed at different areas of prepared enamel.

## MATERIALS AND METHODS

### Tooth specimen preparation

The protocol for this research was approved by the Commission of Medical Ethics of Alexandria University under the file number 0124-03/2020. Twenty-seven sound maxillary premolars were randomly selected for this study. After external debris were removed, the teeth were stored in a 0.2% thymol solution for seven days to destroy any remaining microorganisms. They were then stored in distilled water. Each tooth was mounted individually in an acrylic resin cylinder (14 × 20 mm), 2 mm apical to the cemento-enamel junction. To provide space for the simulated periodontal ligament, a 0.3 mm layer of wax was applied on the roots. The teeth and wax were removed from the acrylic blocks leaving a socket-shaped hole, and polyether adhesive (Polyether Adhesive, 3M ESPE, GmbH, Neuss, Germany) was coated on the roots until it dried fully. Polyether impression material (Impregum Soft, 3M ESPE, GmbH, Neuss, Germany) was then coated on the roots and the tooth was returned in its acrylic block to mimic the periodontal ligament. Any excess was removed using a sharp blade<sup>(8,15)</sup>.

All the materials with their composition are shown in Table 1.

TABLE (1) The restorative materials used in the study.

Brand name	Manufacturer	Composition
IPS e.max CAD	Ivoclar Vivadent, Schaan, Liechtenstein	<ul style="list-style-type: none"> <li>• SiO<sub>2</sub> 60.0 - 65.0 % by wt</li> <li>• Al<sub>2</sub>O<sub>3</sub> 16.0 - 20.0% by wt</li> <li>• K<sub>2</sub>O 10.0 - 14.0 % by wt</li> <li>• Na<sub>2</sub>O 3.5 - 6.5%by wt</li> <li>• Other oxides 0.5 - 7.0% by wt</li> <li>• Pigments 0.2 - 1.0% by wt</li> <li>1 – 5 μm leucite crystals (35–45 % by volume)</li> </ul>
RelyX Veneer	3M Deutschland, Neuss, Germany	<ul style="list-style-type: none"> <li>• Silane-treated ceramic (55-56% by wt)</li> <li>• TEGDMA (triethylene glycol dimethacrylate) (10- 20-% by wt)</li> <li>• BISGMA (bisphenol A-diglycidyl etherdimethacrylate) (10-2-% by wt)</li> <li>• silane treated silica (1-10% by wt)</li> <li>• reacted polycarpolactone polymer (1-10% by wt)</li> <li>• titanium dioxide (&lt;1% by wt)</li> <li>• diphenyliodonium hexafluorophosphate (&lt;0.5 by wt.)</li> <li>Triphenylantimony (&lt;0.5 by wt.)</li> </ul>
Single Bond Universal Adhesive	3M Deutschland, Neuss, Germany	<ul style="list-style-type: none"> <li>• 2-hydroxyethyl methacrylate (15 - 25 % by wt)</li> <li>• BIS-GMMA (Bisphenol A-diglycidyl ether dimethacrylate) (15 - 25 % by wt)</li> <li>• Decamethylenedimethacrylate (5-15% by wt)</li> <li>• Ethanol (10 – 15% by wt)</li> <li>• Silane treated silica ( 5 - 15 % by wt)</li> <li>• Water (10 - 15 % by wt)</li> <li>2-propenoic acid, 2-methyl-, reaction (1-10% by wt)</li> </ul>

### Tooth surface preparation

With a high-speed handpiece and depth limiting and tapered diamond burs of medium grit (Microdont, Monsey, NY, USA), a 0.5-mm depth preparation into the enamel of the buccal surface was performed. A stereomicroscope was used to ensure that no dentin was exposed<sup>(8,15)</sup>.

An extraoral scanner (Vinyl scanner, Smart optics Sensortechnik, GmbH, Bochum, Germany) was used to scan the tooth surfaces. Twenty-seven ceramic blocks of size C14 (IPS e.max CAD, Ivoclar Vivadent, Schaan, Liechtenstein) were used in the milling of the microdiscs, with three microdiscs being milled out of a single block (27x3). A total of 81 ceramic microdiscs of cylindrical shape (n=9), three on each of the 27 teeth, were designed using Exocad

software (Exocad GmbH, Darmstadt, Germany)<sup>(16)</sup>. Each microdisc had dimensions of 1 mm in diameter and 1 mm in height. Various cement film thicknesses were assigned to each group: group A=20 μm, group B=40 μm, and group C=100 μm. During the design of the discs, the periphery of the discs had no cement gap, which was only at the center of the discs, to ensure a definite seat on the tooth surface. Blocks were then milled using the CEREC inLab MC XL CAD/CAM milling machine (Sirona Dental Systems, GmbH, Bensheim, Germany)<sup>(17)</sup>. The partially crystallized veneers were then crystallized using Programat p310 (Ivoclar Vivadent, Schaan, Liechtenstein). The microdiscs were cemented onto three different areas of the buccal surface: coronal (c), middle (m), and cervical (v) thirds.

### Surface treatment of ceramic microdiscs

After the fabrication of the specimens, 8% hydrofluoric acid (Dentobond etching gel, Itena, Villepinte, France) was used to etch the bonded surface of the microdiscs for 60 seconds. The acid was then rinsed away with a water spray and air-dried with oil- and water-free compressed air. The etched ceramic surface was then silanated (RelyX ceramic primer, 3M ESPE, St. Paul, MN, USA). The primer was allowed to react for 60 seconds, then air-dried for 2-5 seconds. The intaglio surfaces of the microdiscs were coated with a single layer of adhesive, which was then thinned with an air syringe for 5 seconds. The surfaces of the microdisc are so small that it was necessary to use a digital light microscope (Inskam 307, Shenzhen, China) to view their fitting surfaces before the application of conditioning materials and the adhesive<sup>(8)</sup>.

### Surface treatment of prepared enamel surface

Phosphoric acid 37% (N-etch gel, Ivoclar Vivadent, Schaan, Liechtenstein) was used to etch the central area of the buccal surface where the microdiscs were to be cemented. The etching gel was left for 15 seconds, rinsed for 10 seconds and excess water was removed with a cotton pellet<sup>(18)</sup>. Two layers of universal adhesive (Single Bond Universal Adhesive, 3M ESPE, St. Paul, MN, USA) were applied onto the etched enamel surfaces and rubbed for 20 seconds. The adhesive was then evenly distributed using an air spray to form a shiny and homogeneous film<sup>(8)</sup>.

### Cementation of microdiscs

The microdiscs were cemented on the coronal, middle, and cervical thirds of the buccal surface using a light-cure resin cement (RelyX Veneer, 3M ESPE, St. Paul, MN, USA). A tweezer was used to set the microdiscs on the prepared buccal surface. They were then fully polymerized for 40 seconds using a light-emitting diode unit with a light intensity of 1200 mW/cm<sup>2</sup> (Elipar™ FreeLight

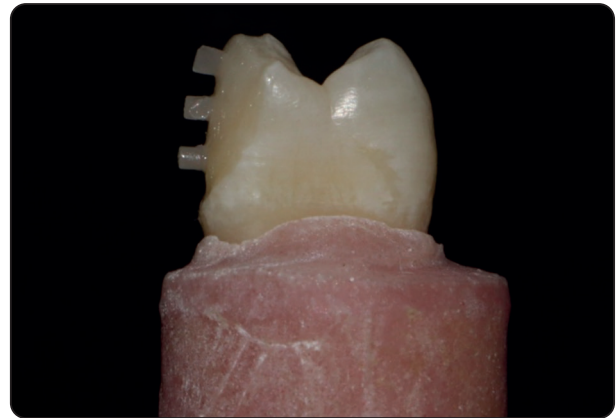


Fig. (1): Ceramic microdiscs cemented on the coronal, middle, and cervical thirds of the buccal surface (proximal view).

2, 3M ESPE, St. Paul, MN, USA) (Fig. 1). Artificial aging was performed by thermocycling for 2,500 cycles in water baths with temperatures between 5 and 55°C with a dwell time of 15 seconds and 5 seconds transfer time (Julabo, Seelbach, Germany), simulating approximately three months of aging<sup>(8)</sup>.

### Microshear bond strength test

Microshear bond strength was then performed using a mono-beveled chisel mounted on a universal testing machine (5ST Tinius Olsen, Redhill, UK). The chisel fell at the tooth and microdisc interface at a cross-head speed of 0.5 mm/min<sup>(8)</sup>.

A stereomicroscope (SZ114STR, Olympus, Tokyo, Japan) at a magnification of 50× was used to examine the tooth surface to assess the mode of failure. Failures were classified as cohesive if greater than 75% of luting resin remained on the tooth surface, adhesive if less than 25% of the luting resin remained on the tooth surface, or mixed if the remaining luting resin was between 25% and 75% on the tooth surface (Fig. 2)<sup>(8,19)</sup>.

### Statistical analysis

Data was analyzed using IBM SPSS statistical software (version 25; IBM Corporation, Armonk, NY, USA). Significance level was set at 0.05.

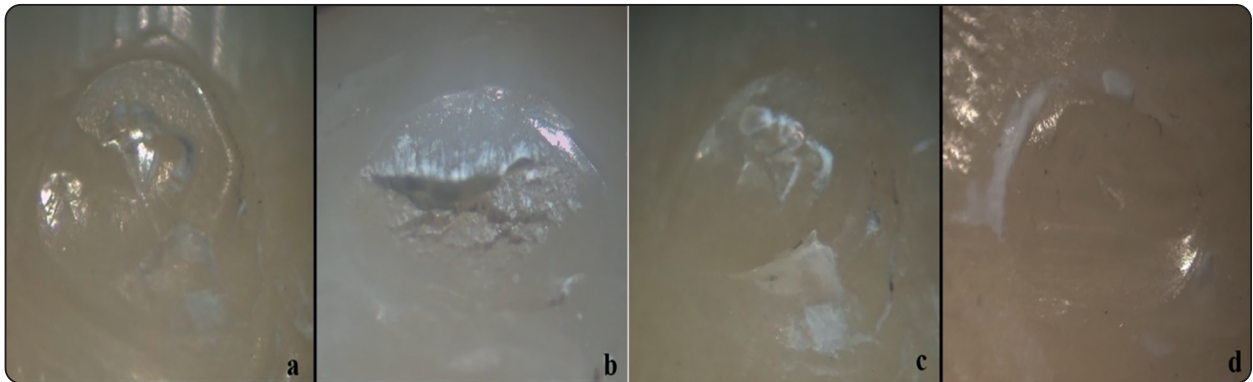


Fig. (2): Stereomicroscope images at 50× magnification. (a) Cohesive failure at ceramic and cement interface, (b) Cohesive failure in enamel and ceramic, (c) mixed failure, (d) adhesive failure.

Normality was checked using Shapiro-Wilk test and box plot. Microshear bond strength was normally distributed and presented using mean and standard deviation. Type of fractures was presented using count and percent. Differences between groups regarding the microshear bond strength were assessed using one-way ANOVA followed by Tukey's post hoc test while comparisons between the coronal, middle and cervical thirds were done using repeated measures of ANOVA followed by post hoc test with Bonferroni correction. The mode of failure was compared using chi-square test.

TABLE (2) : Comparison of  $\mu$ SBS mean  $\pm$  standard deviation (MPa) between the study groups at different tooth areas

	Group A: 20 Microns (n=9)	Group B: 40 Microns (n=9)	Group C: 100 Microns (n=9)	Test (p value)
	Mean (SD)			
Coronal (c)	34.91 (6.72)	36.08 (4.11)	16.89 (2.72)	44.955 (<0.0001*)
Middle (m)	29.08 (6.55)	30.72 (4.85)	15.06 (2.62)	27.227 (<0.0001*)
Cervical (v)	17.41 (5.91)	21.94 (4.15)	10.97 (1.01)	15.387 (<0.0001*)
Test (p value)	27.178 (<0.0001*)	19.798 (<0.0001*)	28.501 (<0.0001*)	
Pairwise comparisons	$P_1=0.294$ $P_2<0.0001^*$ $P_3=0.001^*$	$P_1=0.209$ $P_2=0.001^*$ $P_3=0.006^*$	$P_1=0.011^*$ $P_2=0.001^*$ $P_3=0.007^*$	

\*Statistically significant different at  $p$  value  $\leq 0.05$  P1: Comparison between coronal and middle, P2: Comparison between coronal and cervical, P3: Comparison between middle and cervical

## RESULTS

The mean values and standard deviations of the microshear bond strength test are shown in Table 2 and Figure 3. The group with the 40- $\mu$ m cement thickness had the highest  $\mu$ SBS in all three areas of the tooth.

The highest  $\mu$ SBS was observed in the microdiscs cemented on the coronal third with a 40- $\mu$ m cement thickness (group Bc), yet one-way ANOVA ( $p \leq 0.05$ ) indicated that it was insignificant compared to that of microdiscs cemented with a 20- $\mu$ m thickness in the same area (group Ac).

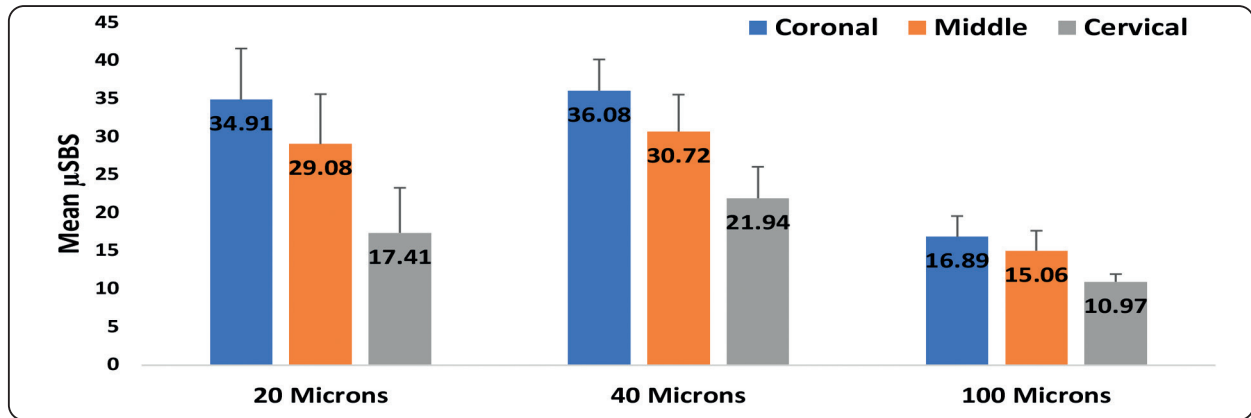


Fig. (3): Comparison of μSBS between the study groups at different tooth areas.

Cement thicknesses of 20 and 40 μm did not affect the bond strength significantly when the microdiscs were cemented on the coronal or middle thirds. However, an increase in cement thickness to 100 μm caused a statistically significant decrease in bond strength between the coronal and middle thirds. Adhesion to the cervical region was the poorest for all cement thicknesses. Microdiscs cemented with a 100-μm film had the lowest values in all areas of the buccal surface. Pairwise comparison between the study groups regarding the μSBS at different tooth areas are shown in Table 3.

Regarding the mode of failure, there was no significant difference in the overall results. However, the coronal and middle thirds showed a predominance of cohesive failures, whereas adhesive failures were mostly observed in the cervical third (Fig. 4) (Table 4).

TABLE (3) Pairwise comparison between the study groups regarding the μSBS at different tooth areas

	Groups	Compared to	P value
Coronal	20 Microns	40 Microns	0.865
		100 Microns	<0.0001*
	40 Microns	100 Microns	<0.0001*
Middle	20 Microns	40 Microns	0.763
		100 Microns	<0.0001*
	40 Microns	100 Microns	<0.0001*
Cervical	20 Microns	40 Microns	0.078
		100 Microns	0.009*
	40 Microns	100 Microns	<0.0001*

Table (4) Mode of failure among the study groups at different tooth areas

		20 Microns (n=9)	40 Microns (n=9)	100 Microns (n=9)	Test (p value)
Coronal	Adhesive	0 (0%)	0 (0%)	0 (0%)	0.355 (0.837)
	Cohesive	6 (66.7%)	6 (66.7%)	7 (77.8%)	
	Mixed	3 (33.3%)	3 (33.3%)	2 (22.2%)	
Middle	Adhesive	1 (11.1%)	1 (11.1%)	2 (22.2%)	1.727 (0.786)
	Cohesive	5 (55.6%)	5 (55.6%)	4 (44.4%)	
	Mixed	3 (33.3%)	3 (33.3%)	3 (33.3%)	
Cervical	Adhesive	7 (77.8%)	6 (66.7%)	6 (66.7%)	2.391 (0.664)
	Cohesive	0 (0%)	1 (11.1%)	0 (0%)	
	Mixed	2 (22.2%)	2 (22.2%)	3 (33.3%)	



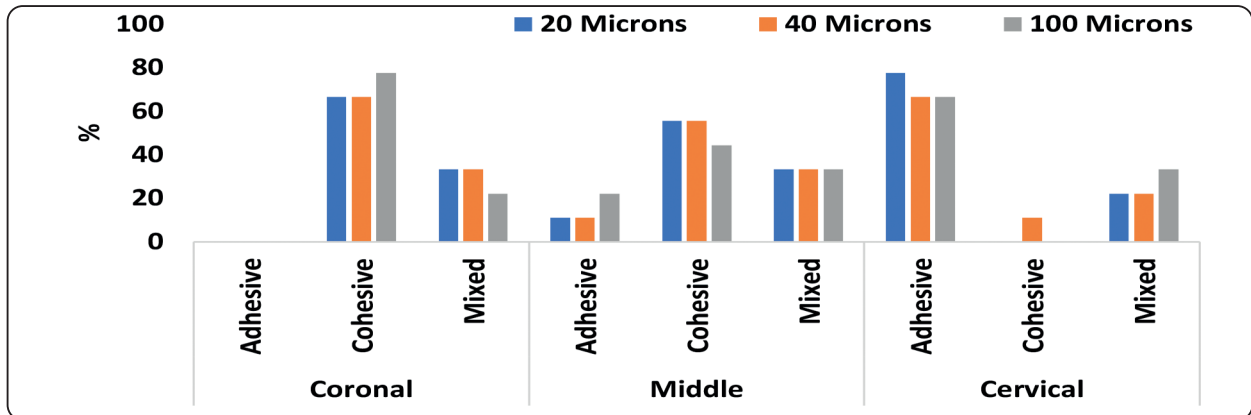


Fig. (4): Mode of failure among the study groups at different tooth areas.

## DISCUSSION

In this study, a curved enamel surface, as opposed to a flat surface which is common in *in vitro* microshear bond strength tests, was chosen to mimic clinical situations. Shear forces of mastication do not fall on flat surfaces, and although a curved surface would engender a more complex series of forces on the ceramic-tooth interface, this technique would also provide greater insight on the clinical performance of lithium disilicate, despite being a laboratory study. Maxillary premolars were chosen as they are often subjected to high shear and tensile stresses during mastication. A 0.5 mm enamel reduction was performed to imitate a preparation that would be clinically done for veneers. The cylindrical shape and dimensions of the microdiscs were chosen following a previous study<sup>(16)</sup>.

The microshear bond strength test is essentially a traditional shear bond test but on a much smaller scale. Bonding to a minuscule surface area prevents any discrepancies in enamel thickness that may occur during full preparations, thus providing more accurate results. Moreover, a smaller ceramic specimen would ensure that the cement gap provided was uniform throughout the intaglio surface, which is not always the case in full restorations. Venturini *et al.* found that the cement gap set digitally was not always equal to the resulting gap after fabrication of the restoration<sup>(20)</sup>.

Enamel has one of the most complex morphologies within the human body. Its rod and interrod structures, often described as having a “keyhole” appearance, are the source of its mechanical performance<sup>(14)</sup>. Enamel rod orientation varies greatly along the surface of the tooth. The cervical portion of the crown enamel exhibits a disturbed prism arrangement, lacking the keyhole appearance of coronal enamel. Moreover, a thicker layer of aprismatic enamel is present cervically. Aprismatic enamel is highly mineralized, with little to no prism boundaries where inorganic matrix should be present. Its very high mineral content makes acid etching difficult and contributes to the considerably lower bond strengths<sup>(14)</sup>. Previous studies have shown that resin tags formed in the cervical region were shorter and less numerous than those in the mid-coronal region<sup>(14,20)</sup>. Clinically, cervical enamel is also more prone to dentin exposure as it is much thinner than coronal and middle third enamel. Adhesion to dentin would exhibit even lower bond strengths than those in the present study.

Regarding the role of cement thickness on the bond strength of lithium disilicate to enamel, an increase in thickness seemed to only worsen the already low bond strengths of cervical enamel. The sparse and short resin tags formed would retract even more due to high polymerization shrinkage

generated by a thicker cement. The results in the failure modes of this study clearly show a defect in the adhesion at the tooth-cement interface as there is a majority of adhesive failures.

In contrast to the cervical region, the coronal and middle regions of enamel have a thin aprismatic layer that is easily removed by acid etching, exposing the keyhole appearance, or microporosities, of prismatic enamel<sup>(21)</sup>. Prismatic enamel is perpendicular to the enamel surface, thus facilitating resin penetration. The cement film thickness of 40  $\mu\text{m}$  exhibited the highest mechanical performance in all three areas of the tooth. However, cement thickness did not have a significant impact on the bond strengths of the coronal and middle thirds at 20 and 40  $\mu\text{m}$ , presumably because of their similarity in enamel morphology and resin tag penetration. Nevertheless, the deleterious effect of a thicker film appeared at 100  $\mu\text{m}$ , where a significant difference in bonding can be observed between the coronal and middle thirds. Even when on a morphologically favorable area for bonding, the bond strength significantly decreased due to a thicker cement thickness.

During the examination of failure modes, most of the cohesive failures were between the cement and ceramic interfaces. However, a few specimens showed cohesive failures within the enamel, indicating very high bond strength to the tooth substrate, but also possibly the initiation of cracks within the enamel due to polymerization shrinkage that occurred within the deep resin tags<sup>(22)</sup>.

## CONCLUSION

Within the limitations of this study, the following conclusions can be made:

- A thicker cement film thickness in the cervical area of the tooth greatly decreases  $\mu\text{SBS}$  of lithium disilicate, while microdiscs cemented coronally with a thinner film showed better adhesion.

- Adhesion in the cervical area is not improved by lower or higher cement thicknesses.
- A thicker cement film only negatively impacts the coronal and middle thirds of a tooth surface when it reaches 100  $\mu\text{m}$ .

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## Conflicts of interest

Authors declare no conflicts of interest.

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