Original Article

Comparative Evaluation of Microtensile Bond Strength in Three Different Dentin Luting Agents: An *In vitro* Study

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Abstract

Aim: Long-term clinical success on indirect restorations is largely determined by bonding efficiency of the luting agent, with adhesion to dentin being the main challenge. Therefore, aim of this study was to assess the microtensile bond strength when using flowable resin composite, preheated resin composite and dual self-adhesive resin cement as dentin luting agents. **Materials and Methods:** Occlusal thirds of molar teeth were cut and randomly divided into 3 groups to be cemented: RelyX™U200, Filtek™ Z250 XT- preheated to 70° and Filtek Flow™ Z350XT. They were then thermocycled 5000 times between 5+/-2°C and 55+/-2°C. Subsequently, 10 microbars per group were prepared. The 30 samples were placed in saline solution for 24 hours at room temperature prior to microtensile test. This was performed with a digital universal testing machine at a crosshead speed of 0.5 mm/min. The bond strength values obtained were analyzed in Megapascals (MPa). Measures of central tendency such mean and measures of dispersion such standard deviation were used. In addition, the Kruskall Wallis non-parametric test with Bonferroni post hoc test was applied, considering a significance value of 5% ($P < 0.05$), with type I error. **Results:** The dentin microtensile bond strengths of preheated resin composite, flowable resin composite and dual self-adhesive cement were 6.08 ± 0.66 Mpa, 5.25 ± 2.60Mpa and 2.82 ± 1.26Mpa, respectively. In addition, the preheated resin composite exhibited significantly higher microtensile bond strength compared to the dual self-adhesive cement ($P < 0.001$). While the flowable resin composite showed no significant difference with the dual self-adhesive cement ($P = 0.054$) and the preheated resin composite ($P = 0.329$). **Conclusion:** The microtensile bond strength in dentin was significantly higher when using a preheated resin composite at 70°C as a luting agent compared to dual self-adhesive cement. However, the preheated resin composite showed similar microtensile bond strength compared to the flowable resin composite.

Keywords: *Dentistry, flowable resin, luting agent, microtensile bond strength, preheated resin, resin composite*

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Introduction

Indirect restorations based on conventional resin composites are an affordable alternative with good esthetic results. Therefore, many dentists prefer them because of their better mechanical performance

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and significant reduction of shrinkage during polymerization compared to a direct restoration,[1-5] since the direct technique has a volumetric shrinkage rate of 1% to 6%, depending on the composition of the resin and curing conditions, which would be reduced when performing an indirect technique.[4] They also have a lower incidence of fractures compared to ceramics, since these have shown up to 20% fracture rates in studies clinical Class I and II cavities.[3]

Several authors point out that one of main reasons for failure of indirect restorations is due to lack of sealing at the tooth-restoration interface, since the shrinkage produced by polymerization of luting agent would cause the formation of marginal spaces or microgaps around inlay, allowing bacterial infiltration and entry of food debris, which would generate secondary caries.[5-7] Therefore, it is considered that long-term clinical success of indirect restorations is largely determined by bonding efficiency of the luting agent.[5,6]

In order to increase the bond strength to dental substrate, some modifications have been made to bonding agents regarding their chemical composition, bonding mechanism, number of steps and application technique. As a result, there are several generations of bonding agents available on the market.[8]

Conventional composite resins continue to be one of the most widely used materials in dentistry, being used in direct and indirect restorations, either as a restorative material or as a luting agent.[9,10] The composition of composite resins is based on an organic matrix with a mixture of monomers such as Bis-GMA (Bisphenol-A-Glycidyl Methacrylate), TEGDMA (Triethylene Glycol Dimethacrylate), UDMA (Urethane Dimethacrylate), HEMA (Hydroxy Ethylmethacrylate), Bis-EMA (Bisphenol A Polyethylene Glycol Diether Dimethacrylate), fillers such as silica, quartz or ceramic glass and a photoinitiator such as camphorquinone, BAPO (Bisacyl Phosphine Oxide) or others, resulting in macroparticulate, microparticulate, nanoparticulate and hybrid composite resins containing particles of different sizes.[9,11,12] On the other hand, when referring to resinous cements, the composition varies, having two main differences: the proportion of monomers used in the mixture and the amount of filler, which confers lower viscosity for higher flow, but at the same time reducing its mechanical properties compared to conventional resin composites.[9,12]

Due to great versatility of resin composites and their superior mechanical properties, alternative techniques have been used to reduce their viscosity and use them as luting agent. The best known is thermoplastic technique, in which resin becomes less viscous when heated due to increased agitation of molecules, providing sufficient viscosity for bonding indirect restorations, similar to resin cements.[9,11] Consequently, preheated composite could be used as luting agent without losing its mechanical strength properties, giving the advantage of greater color shade availability and lower cost, in addition to being able to preheat the same resin syringe up to 20 times without affecting its mechanical properties. However, there is currently no consensus on whether preheated resin composite would increase or decrease the bond strength between the restorative material and the substrate. In addition, there is still some controversy over the thickness of the film formed from it, with some authors claiming that inlay sits well in the cavity, while others do not.[9,13,14]

Another alternative as a luting agent is composite resin as it has lower viscosity due to its low amount of filler and particle size, similar to conventional composite resins. These characteristics give as an advantage greater color stability over time, better diffusion of the material, wide range of colors for different clinical situations, in addition to polymerization shrinkage and film thickness similar to resinous cements. On the other hand, due to its low inorganic filler, it has the disadvantage of higher polymerization shrinkage. However, its low modulus of elasticity would allow shrinkage to take place with little stress, reducing the formation of micro-gaps.[10,15-17]

Dual resinous cement, which possesses advantageous characteristics of light-curing and self-curing cements, is also used to cement indirect restorations because of its low solubility, low viscosity, clinically acceptable film thickness, better mechanical properties than conventional cements, good adhesion when used as a bonding agent and less microleakage compared to other luting materials. As a disadvantage, it has been suggested that early vitrification (formation of polymeric networks) induced by light activation could interfere with autopolymerization, thus compromising the degree of conversion. In addition, it has been pointed out that insufficient light exposure could result in incomplete polymerization, with a lower level compared to self-curing resinous cements.[6,18]

Several studies report that bonding to enamel is achieved in a firm and durable manner with relative ease. However, bonding to dentin has been considered more difficult and less predictable due to its heterogeneous nature containing hydroxyapatite deposited on a mesh of hydrophilic collagen fibers, representing a great challenge for bonding agents.[18,19] Since bonding agents are of utmost importance for preservation of the restoration, it has been suggested to carry out more *in vitro* research on adhesive tests, the most widely used being microtraction or microtensile tensile strength.[19,20] In view of the above, the constant evolution of dental materials leads to rethinking of techniques, materials and treatments in order to provide a quality restoration to the patient.

Therefore, objective of the present study was to evaluate the *in vitro* microtensile adhesive strength when using flowable resin composite, preheated resin composite and dual self-adhesive resin cement as dentin luting agents.

Materials and Methods

Bioethical considerations

This research respected the bioethical principles for medical research with human beings of the Declaration of Helsinki. Likewise, it was approved by the Ethics and Research Committee of the Faculty of Stomatology of the Universidad Privada San Juan Bautista with official letter No. 436-2021- CIEI-UPSJB. The teeth obtained for the present investigation were donated by the patients, with prior informed consent.

Type of study and delimitation

This experimental *in vitro*, cross-sectional and analytical study was carried out at Stomatology School of the Universidad Privada San Juan Bautista and at the High Technology Laboratory Certificate (ISO/IEC Standard: 17025), Lima - Peru; from July to October 2021. This study considered the CRIS Guidelines (Checklist for Reporting In-vitro Studies).[21]

Sample calculation and selection

30 sample units were distributed in three groups under a completely randomized block design without replacement (n = 10), and were calculated based on data obtained in a previous pilot study where mean comparison formula was applied considering a significance level (α) = 0.05, a statistical power (1-β) = 0.80 with variances S12 = 0.41 and S22 = 1.56, and a mean difference equal to 1.3 Mpa. The experimental groups, according to luting agents employed were:

- Group 1: RelyX™U200 Dual Self-Adhesive Resinous Cement (3M ESPE, St. Paul, MN, USA)
- Group 2: Filtek ™ Z250 XT- Resin composite preheated to 70° (3M ESPE, St. Paul, MN, USA).
- Group 3: Filtek Flow ™ Z350XT Flowable resin composite (3M ESPE, St. Paul, MN, USA).

Study variables

Variables included in the present study were: type of luting agent and bond strength.

Sample characteristics and preparation

Teeth were extracted during the last 3 months prior to experiment, removing remains of soft tissue or bacterial plaque with a dental ultrasonic scaler (DTE D5 LED, Woodpecker, Guilin, Guangxi, China). They were then stored in 0.9% sodium hypochlorite solution. Subsequently, they were divided into three groups to cut the occlusal third with a rotary tool (DREMEL® 300 Series, Mt. Prospect, Illinois, U.S) and a low-speed water-cooled diamond cutting disc. In addition, a standard mold was made to produce 6 x 6 mm resin blocks with Filtek™ Z250 XT resin composite. Prior to cementation, all resin blocks were micro-sandblasted (MicroJato, Bio-art, Sao Carlos, SP, Brazil) for 15 seconds. Cementation was then performed according to 3 experimental groups: [Figures 1 and 2]:

- **Group 1:** Dual RelyX™U200 Self-Adhesive Resin Cement. It was cemented according to manufacturer's instructions. Surface was cleaned with pumice stone and resin cement was placed on the resin block. The resin cement was set on the surface exerting pressure to eliminate the excess with the help of a microbrush and then light cured with an third generation LED lamp (Valo - Ultradent, South Jordan, UT, USA) with an intensity of 1200 mW/cm2 for 20 seconds.
- **Group 2:** Filtek™ Z250 XT- resin composite preheated to 70°. A 37% acid etch with Scotchbond™ Etchant™ was performed (3M ESPE, Maplewood,

Figure 1: Materials used as luting agents

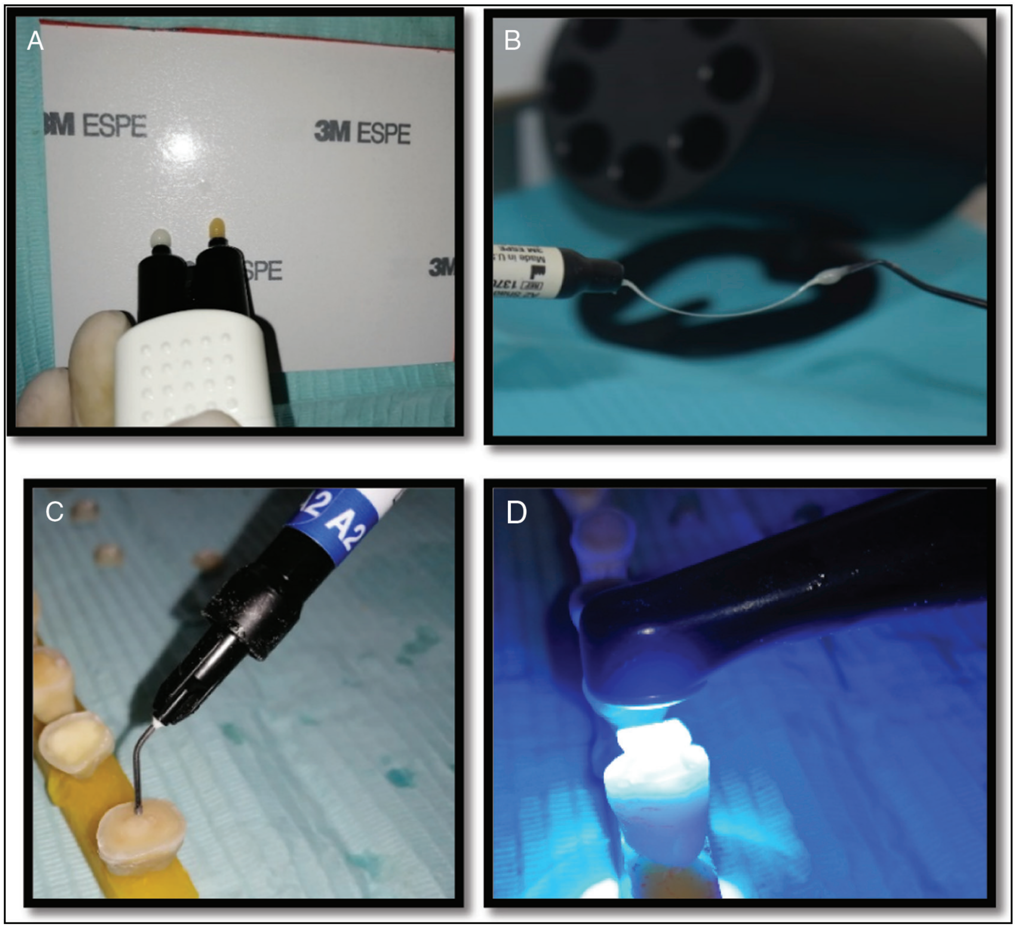


Figure 2: A: Dual RelyX™U200 Self-Adhesive Resin Cement Preparation. B: Z250XT resin composite preheating. C: Placement of Z350XT Flowable resin composite. D: Light curing of samples with third generation LED curing lamp (Valo - Ultradent, South Jordan, UT, USA)

Minnesota, USA), for 15 seconds, then washed with water for 10 seconds and dried the excess moisture with cotton. Then, a single layer of Adper™ Single Bond 2 adhesive (3M ESPE, Maplewood, Minnesota, U.S.) was applied on sample, evaporating the solvent with gentle air flow for 3 seconds and light cured with an LED lamp at an intensity of 1200 mW/cm2 for 20 seconds. Resin composite was preheated in an AR Heater (Zhengzhou, Henan, China) at 70°C [Figure 3] for 10 minutes and then cemented. The material was placed in the resin block, settled on surface exerting pressure to eliminate excess, which was removed with microbrush, and light cured with LED lamp at an intensity of 1200 mW/cm2 for 20 seconds.

- **Grupo 3:** Filtek Flow™ Z350XT Flowable Resin Composite. A 37% acid etch was performed with Scotchbond™ Etchant for 15 seconds, then washed with water for 10 seconds and dried the excess moisture with cotton. Then a single layer of Adper™ Single Bond 2 adhesive was applied on sample, evaporating the solvent with gentle air flow for 3 seconds and light cured with an LED lamp at an intensity of 1200 mW/cm2 for 20 seconds. Then, the material was placed in the resin block, settled on surface exerting pressure to eliminate the excess, which was removed with microbrush, and light cured with LED lamp at an intensity of 1200 mW/cm2 for 20 seconds.

Subsequently, 5000 thermocycles between 5+/-2°C and 55+/-2°C were applied to all study samples. For better handling, teeth were placed on an acrylic base to obtain microbars. Horizontal and vertical cuts were made using a low speed water-cooled diamond cutting disc, changing the disc every 5 cuts. The dimensions of microbars were 1 mm × 1 mm × 8 mm[22] obtaining 10 bars per group [Figure 4]. Measurements were made with a digital vernier caliper (Mitutoyo, Kawasaki, Kanagawa, Japan).

Microtensile test

Once the 30 samples were obtained, they were placed in saline solution for 24 hours at room temperature prior to microtensile testing. The tests were performed using

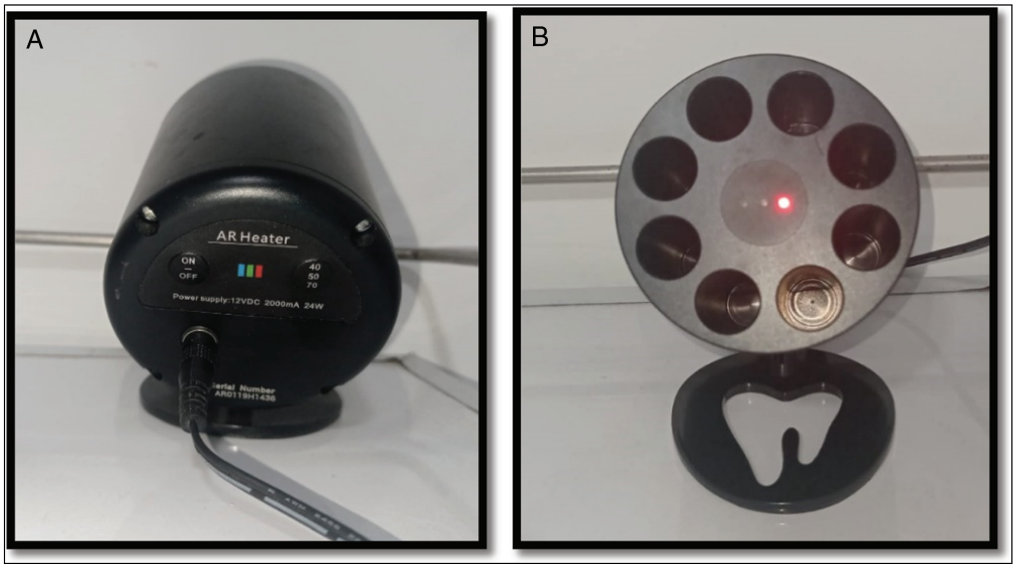


Figure 3: A: Rear side of heater with temperature indicator. B: Front side of heater (red color indicates 70°C)

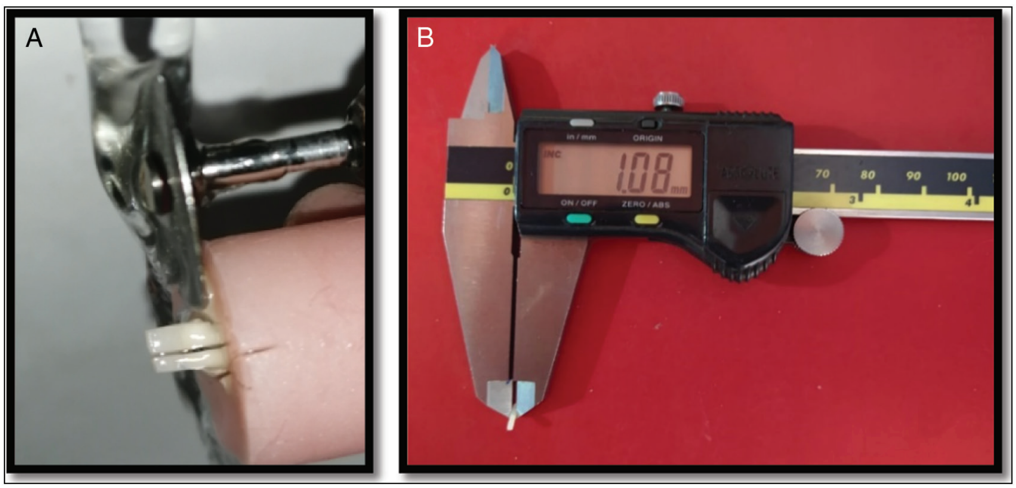


Figure 4: A: Cutting of microbars. B: Measurement of microbars

a digital universal testing machine (CMT-5L Liangong, Shandong, China) with a software digital (Smart Test) at a crosshead speed of 0.5 mm/min [Figure 5]. The bond strength values obtained after the test were analyzed in Megapascals (MPa).

Statistical analysis

The data collected were entered in a Microsoft Excel 2019® file and subsequently imported for statistical analysis by the SPSS program (Statistical Package for the Social Sciences Inc. IBM, NY, USA) version 24.0. For descriptive analysis, measures of central tendency such mean and measures of dispersion such standard deviation were used. For comparative analysis, the Shapiro Wilk normality test and Levene's homoscedasticity test were previously performed. With the results, the statistical decision to use the nonparametric Kruskall-Wallis test with the Bonferroni post hoc test was made, considering a significance value of 5% ($P < 0.05$), with a type I error.

Results

The preheated resin composite as dentin luting agent presented higher values of microtensile bond strength with a mean of 6.08 ± 0.66 Mpa, while the dual self-adhesive cement presented the lowest values with a mean of 2.82 ± 1.26 Mpa. [Table 1 and Graph 1].

When comparing the microtensile bond strength, it was noticed that there were significant differences ($P < 0.001$) between the three dentin luting agents, with the preheated resin composite obtaining less dispersed values (RIQ = 0.99) [Table 2].

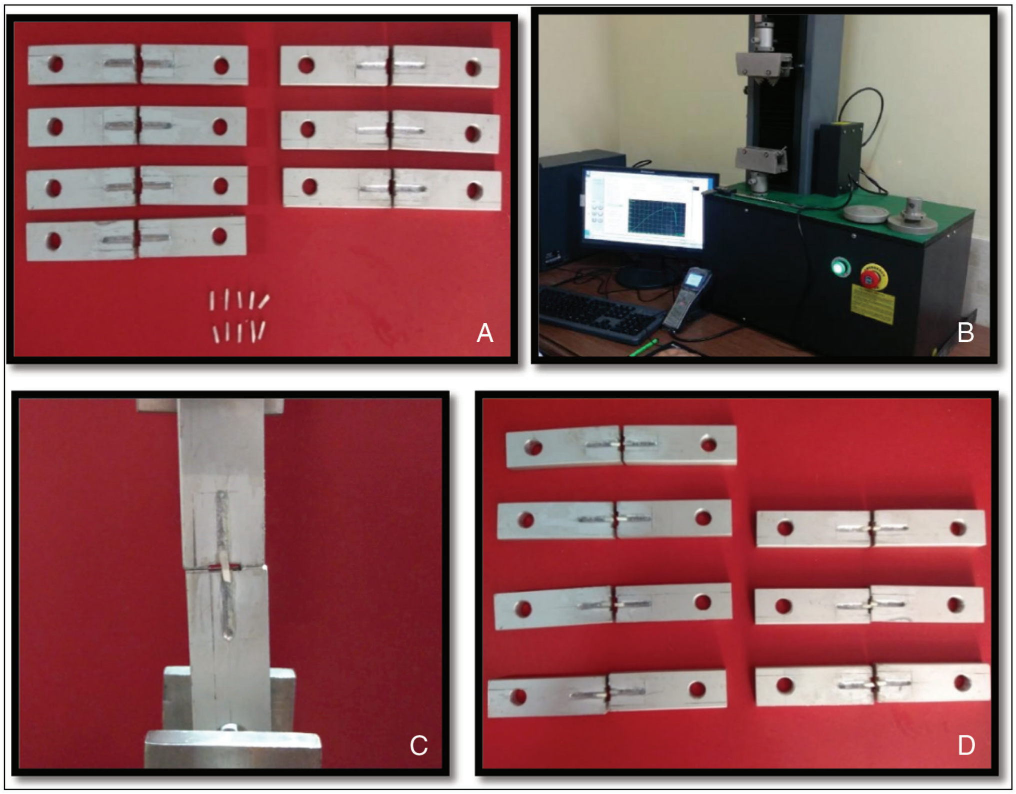
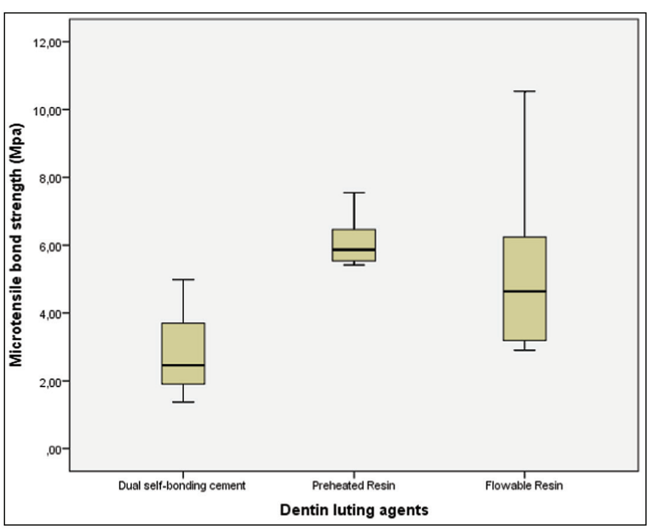


Figure 5: A: Samples prior to microtensile. B: Microtensile Universal Testing Machine. C: Microtensile test. D: Samples after microtensile

Table 1: Microtensile bond strength (Mpa), according to luting agent

Luting agent	n	Median	Mean	SD	SE	95% CI		Min	Max
						LL	UL		
Dual self-bonding cement	10	2.46	2.82	1.26	0.40	1.92	3.72	1.37	4.98
Preheated Resin	10	5.87	6.08	0.66	0.21	5.61	6.56	5.42	7.55
Flowable Resin	10	4.64	5.25	2.60	0.82	3.39	7.11	2.90	10.54

Mpa: Megapascals, n: sample, SD: Standard Deviation, SE: Standard Error, Ll: Lower Limit, UL: Upper Limit, CI: Confidence Interval, Min: Minimum, Max: Maximum



Graph 1: Distribution of the microtensile bond strength values, according to the dentin luting agent

As dentin luting agent, preheated resin composite presented significantly higher microtensile bond strength in comparison to dual self-adhesive cement ($P < 0.001$). While the flowable resin composite did not present significant differences with the dual self-adhesive cement ($P = 0.054$) and the preheated resin ($P = 0.329$), respectively [Table 3 and Graph 2].

Discussion

Bonding to dentin continues to be a challenge, compared to enamel, due to its hydrophilic nature. In this sense, in order to achieve success when cementing an indirect restoration, it is necessary to use materials that present greater bonding strength to dentin.[18,19,23] Currently there are different adhesive materials and different application techniques. Therefore, the present study aimed to evaluate the microtensile bond strength *in vitro*, using

Table 2: Comparison of microtensile bond strength (Mpa) between the three luting agents

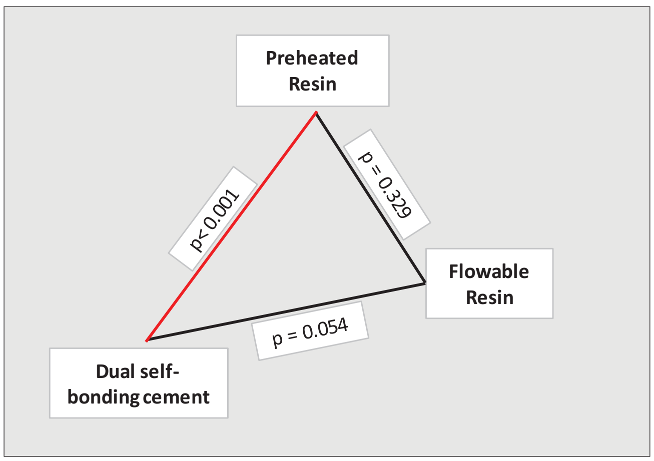
Luting agent	n	Median	IQR	p-value	p-value	p-value
Dual self-bonding cement	10	2.46	2.16	0.113	0.008[b]	<0.001[c]
Preheated Resin	10	5.87	0.99	0.106		
Flowable Resin	10	4.64	3.75	0.038[a]		

n: sample, IQR: interquartile range; [a]Shapiro-Wilk normality test, no normal distribution (p<0.05); [b]Levene homoscedasticity test, non-homogeneous variances (p<0.05); [c]Kruskal-Wallis test, significant differences (p<0.05)

Table 3: Pairwise comparison of the microtensile bond strength variable, according to luting agent

Luting Agent	Flowable Resin	Preheated Resin
Dual self-bonding cement	p = 0.054	p< 0.001*
Preheated Resin	p = 0.329	-

*Based on Bonferroni adjustment test, significant differences (p<0.05)



Graph 2: Statistical comparisons between luting agents

flowable resin composite, preheated resin composite and dual self-adhesive resin cement, as a dentin luting agent. It was obtained that the preheated resin composite resin presented significantly higher values in microtensile bond strength with respect to the dual self-adhesive cement, and higher values than the flowable resin composite, although not significantly. These results were in agreement with those reported by Goulart *et al*[24] and Ugarte *et al*.[25]

However, this disagrees with Görüs,[7] who mentions that strength values of adhesive cements decrease after preheating, which could be due to the composition of the material used, since it contained fiber inside the filler and preheating could have caused its structural rupture, reducing the bonding values. Likewise, Morais *et al*[26] have reported that effectiveness of preheating depends on the resinous product used. It has also been reported that preheating of resinous composites decreases their viscosity, reduces film thickness and improves marginal sealing. In addition, it has been reported that there is between 10 and 15 seconds of ideal working time when the temperature and viscosity in the resin composite are still optimal, so it should be applied quickly, knowing that after removing it from the heating equipment, its temperature decreases about 50% in two minutes.[7,14,25]

Urcuyo *et al.* mentioned that preheating the resin to 60°C increases the conversion of monomers, originating an increase also in molecular mobility. With higher conversion there is greater crosslinking and the free space of polymers is reduced, improving their mechanical properties.[27] This allows to deduce that, if the temperature increases, the mechanical properties as well. Some reports state that increasing the temperature of resin composite between 54° and 68°C would not damage the pulp chamber,[27] since preheating to 68°C increases the pulp temperature between 0.8 to 1.2°C. To cause pulp damage the temperature should be increased by 5.5°C.[28,29] For this reason, in the present study it was decided to preheat resin to 70°C.

As for dual self-adhesive cement resin (RelyX U200), it presentsphosphoricacidesterandmodifiedmultifunctional methacrylate monomers, which have high affinity for the minerals in substrate, which penetrate the dentin creating micromechanical retention and chemical bonding with the calcium ions of hydroxyapatite.[30,31] This suggest that this material would present a higher microtensile bond strength. However, Bulut *et al.* mentioned that this composite would contain phosphoric acid with a very low pH which would not help the chemical bonding with dentin, resulting in inadequate demineralization and weak formation of the hybrid layer,[32] which would explain the low bond strength obtained in the present study. On the other hand, the hydrophilic monomers present in self-adhesive materials, compared to conventional composites, present a greater tendency to water absorption, which leads to swelling of the matrix and consequent rupture of its polymeric chains. These interactions may weaken the mechanical properties of self-adhesive composites, resulting in significantly reduced bond strength.[33] This further reinforces the poor bond strength obtained.

On the other hand, flowable resin composite showed superior bond strength to dual self-adhesive cement, which may be due to fact that it was placed after acid etching and adhesive application. Acid etching would demineralize the smear layer, exposing the collagen fibers of superficially demineralized dentin, causing increased micromechanical interlocking of the bonding agent and resin inside the dentin surface..[34]

It should be emphasized that compact resin was used to make the luting blocks because it is reported to have less light attenuation compared to other materials. It has also been reported that light attenuation causes reduction in the conversion degree of luting agent and is also associated with reduced mechanical properties.[24] Therefore, in the present study, the use of nanohybrid resin composite was chosen to prevent light attenuation from affecting the mechanical properties of materials tested. Also, the application of 5000 thermocycles between 5+/-2°C and 55+/-2°C to all experimental units was justified in order to simulate the temperature variation that occurs in the oral cavity equivalent to half a year of clinical aging, considering that thermal fluctuations can cause microcracks across the resin interface with the luting agents and this could influence the microtensile bond strength.[35-37]

Results of the present study suggest an alternative for cementation of indirect restorations taking into account the microtensile bond strength of three adhesive systems, allowing to suggest the use of a preheated resin as luting material to ensure greater bond strength to dental substrate. However, these results should be taken with caution, since, as a limitation of this study, it is recognized that results obtained in this *in vitro* study cannot be extrapolated to clinical field. For this reason, it is advisable to conduct randomized clinical trials related to the stated objective. In addition, more comparative studies are needed between the three luting agents used, with a larger sample to ensure the performance of parametric tests and to be able to make statistical inference. Furthermore, it is recommended in future studies to use composites with different filler characteristics, since this could influence the reaction of resin to preheating and therefore its mechanical properties. Likewise, it is suggested to control the variable "time elapsed when placing material", as well as to study the effect of resin preheating on intrapulp temperature.

Conclusion

In summary, with the limitations presented by this *in vitro* study, it can be concluded that the microtensile bond strength in dentin was significantly higher when using a preheated resin composite at 70°C as luting agent compared to dual self-adhesive cement. However, the preheated resin composite showed similar microtensile bond strength compared to flowable resin composite.

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Conflicts of interest

None to declare.

Authors contributions

They conceived the research idea (LCCR), elaborated the manuscript (LCCR, MILC, LACG, CFCR), collected, tabulated the information (LCCR, CLG), carried out the bibliographic search (MILC, CLG), interpreted the statistical results (CFCR, MAV), helped in the development of the discussion (LCCR, HCC, MAV, CFCR), performed the critical review of the manuscript (HCC, LACG, CFCR). All authors approved the final version of the manuscript.

Ethical policy and institutional review board statement

This research respected the bioethical principles for medical research with human beings of the Declaration of Helsinki. Likewise, it was approved by the Ethics and Research Committee of the Faculty of Stomatology of the Universidad Privada San Juan Bautista with official letter No. 436-2021-CIEI-UPSJB.

Patient declaration of consent

Not applicable.

Data availability statement

The data that support the study results are available from the author (Ms. Leonor Castro-Ramirez, e-mail: leonor.castro@upsjb.edu.pe) on request.

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