

Evaluation of Influence of Thermocycling on Shear Bond Strength of Two Different Zirconia Systems Bonded to Dentin Using Resin Cements - An In Vitro Study

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Abstract

Objectives: Information about the retentive strength of luting agents for zirconium oxide-based crowns is limited. The purpose was to determine the ability of selected luting agents to retain a representative zirconium oxide ceramic crown under clinically simulated conditions.

Methods: 56 sound freshly extracted first permanent molars were selected. Teeth were divided randomly into two groups based on the type of zirconia system used. Each group was further subdivided based on the type of resin cement used and each subgroup was further subdivided into two halves based on thermocycling. Shear bond strength was measured using Universal Testing Machine and then the samples were observed under magnification (80X) using a Stereomicroscope to identify the nature of bond failure. Student t test was applied on the data obtained. The log transformation, if required, was applied to normalize the data and $p > 0.05$ was considered significant.

Results : Mean bond strength of Panavia F2.0 with Cercon before and after thermocycling was 9.45 Mpa, 13.45 Mpa and With Ziecon was 9.59 Mpa and 12.37 Mpa respectively. Mean bond strength of Rely X U200 with Cercon before and after thermocycling was 8.10Mpa and 11.81Mpa and with Ziecon was 8.12 Mpa and 10.63Mpa respectively.

Significance: Panavia F2.0 was found to be better presented highly significant results than Rely X U200 with both Zirconia Systems. Thermocycling significantly affects the bond strength of both the resin cements with dentin. There was no significant difference was observed between shear bond strength of two zirconia systems.

Highlights:

- This study revealed that the type and composition of Zirconia systems does not affect the shear bond strength of Zirconia to dentin.
- Composition, type of resin cement, and oral conditions affect the bond strength of Zirconia to dentin.

Introduction

The popularity of all-ceramic dental restorations has increased in recent years. Indeed, many patients are more interested in having esthetic appearance than any other feature of dental service. Dental ceramic restorations have been of increasing interest among dentists and patients, due to their expectations for more natural looking restorations [1]. Despite their good mechanical properties, the Porcelain Fused to Metal (PFM) restorations do not always provide optimal cosmetic values [2].

Zirconia may exist in three crystallographic forms, cubic, tetragonal and monoclinic. All of these phases are variants on the cubic fluorite structure, depending on the addition of minor components. Specific phases are said to be stabilized at room temperature by the minor components such as calcium, magnesium, yttrium or ceria [3].

Adhesion to dentin is obtained by infiltration of resin into etched dentin, producing a micromechanical interlock with partially demineralized dentin, which underlies the hybrid layer or resin interdiffusion zone [4]. Resin cements may be classified as total-etch, self-etch and self-adhesive, depending upon their application to dental tissues. Total-etch resin cement requires the use of phosphoric acid followed by primer and adhesive before the application of resin cement. Self-etch resin cements use an acidic primer, which is not rinsed away, to modify the dental surfaces before bonding. Self-adhesive resin

cements bond to dental tissues without previous application of any bonding adhesive [5].

Bond strength of zirconia ceramic mainly depends on the type of resin cement, primers used for bonding and the intra oral conditions [6]. There are certain indications for use of resin cements such as self-adhesive resin cement is indicated with tooth preparations having adequate taper (2° – 5°), whereas self-etching resin cement is recommended for tooth with a short clinical crown (<3mm) and over-tapered preparations ($>5^{\circ}$) [6].

The bonding of zirconia substructures should be based on both micromechanical and chemical bonding since the micromechanical retention supports chemical bonding and if bonding is based only on chemical compounds, some debonding might happen in moist environments such as in the mouth [7]. Airborne-particle abrasion with Al_2O_3 abrasive particles, tribochemical silica coating or firing glass pearls as a monolayer to the inner surface of zirconia substructure can be used as surface pre-treatments prior to bonding to achieve better bond strength between the zirconia material and resin cement [8].

Intra oral temperature change is another important factor that affects the bonding between the tooth structure and the zirconia core materials. The difference in the coefficient of thermal expansion between tooth structure and restorative

materials might induce degradation of the dentin/restoration interface.

The purpose of this study was to evaluate the effect of two resin cements affecting the bond strength of Zirconia after subjecting the samples to thermocycling. The research hypothesis was that there was no difference in the shear bond strength between two resin cements (Panavia F2.0 and Rely XU200) when bonding with two different zirconia systems (Cercon and Ziecon) after subjecting to thermocycling.

Material and Methods

This study was undertaken in the Department of Prosthodontics, ITS Centre for Dental Research, Ghaziabad; Department of Mechanical Engineering and ITS Engineering College, Greater Noida. 56 sound, freshly extracted first permanent molars were selected for this study. The teeth with caries, restorations, hypoplastic defects or showing signs of attrition were excluded. Any calculus deposits and soft tissue were removed and the teeth were stored in distilled water. The teeth were divided at random into two groups (I) and (II) for Cercon (Degudent, Hanau, Germany) and Ziecon (Jyoti Lab Pvt. Ltd., India) based on the type of zirconia system used and then each group was further subdivided into two groups (I_A), (I_B) and (II_A), (II_B) based on the type of resin cements used for Panavia F2.0 and RelyX U200 and each subgroup further subdivided into two halves (I_{A1}), (I_{A2}), (I_{B1}), (I_{B2}) & (II_{A1}), (II_{A2}), (II_{B1}), (II_{B2}) in which one half were subjected to thermocycling.

A prototype of the Zirconia disc was fabricated using CAD/CAM measuring 4mm in diameter and 3mm in height. This was milled by utilizing the respective Zirconia blocks of 98mm diameter × 10 mm height (*Figure 1*). Sintering (binding together) was conducted as per the manufacturer instructions in a step-wise programmed manner for 4-5 hrs at a temperature between 1400°C to 1600°C.

Teeth selected for the study were kept out of water for one hour before the preparation was started. An indelible pencil was used to delineate the cut on the teeth. The tooth was then embedded in auto-polymerizing acrylic resin block and sectioned through the line of incision using diamond disk. The buccal surface of the mounted molar was made flat in such a way that a uniform thickness of dentine was achieved (*Figure 2*).

Cementation: Group A – Panavia F2.0: Airborne-particle abrasion was done with 50-um aluminium oxide for maximum 15 seconds using 4-5 bar pressure followed by cleaning in ultrasonic bath for 3 minutes. For the surface treatment of the prepared samples, Liquid A and Liquid B of ED PRIMER II were mixed and applied in a single coat on the etched tooth surface, left in place for 30 secs and allowed to dry. Panavia

F2.0 paste A and paste B were mixed thoroughly for 20 seconds, applied on the prepared surface of the zirconia and spread evenly. The zirconia disc with the paste was placed on the prepared tooth surface and held under finger pressure. The cement was then light cured on each side according to manufacturer's instructions. Oxyguard II was applied to the margin of the restoration and left for 3 mins. It was then removed with a cotton roll and water spray (*Figure 3*).

GROUP B - RelyX U200: RelyX U200 was applied on the surface of zirconia. The zirconia disc with the paste was placed on the prepared tooth surface. The cement was then light cured on each side according to manufacturer's instructions.

Thermocycling: After cementation, half of the samples of each group were subjected to thermocycling. Each cast restored tooth was subjected to thermocycling in two different thermal baths with temperature maintained at 5°C and 55°C using distilled water. A temperature regulating button and thermometer were used to monitor temperature fluctuation, if any. The thermocycler was exposed each sample for a period of 15 sec at 5°C & 55°C with 15 seconds interval between each cycle. A total of 5000 temp cycles were carried out for each sample.

The samples were tested for the shear bond strength (SBS) using Universal testing machine (Instron, U.S.A.) at a crosshead speed of 0.5 mm/min until debonding at the cement/adhesive interface occurred (*Figure 4*). The maximum force at which debonding occurs was measured. SBS was calculated in MPa by dividing the maximum force by the cross-sectional area of the bonding surface for each specimen. The formula used for the calculation was as follows: 1) Area of the specimen = $\Pi \times r^2$, 2) Strength = Force per unit area = F / A . The samples were then observed under magnification (80X) using a Stereomicroscope to identify the nature of bond failure, viz. cohesive, adhesive or a combination of both (*Figure 5*).

The data thus obtained was entered into MS Excel spreadsheet and the statistical technique applied were student t-test (to determine the level of significance in the difference of each parameter between two groups.). SPSS version 10 was used to analyse the data. The log transformation, if required, was applied to normalize the data and $p > 0.05$ were considered significant.

Results

A statistically significant ($p > 0.05$) difference in mean SBS was observed while comparing two resin cements (Panavia F2.0 and Rely X U200). Panavia F2.0 presented highly significant results than Rely X U200.

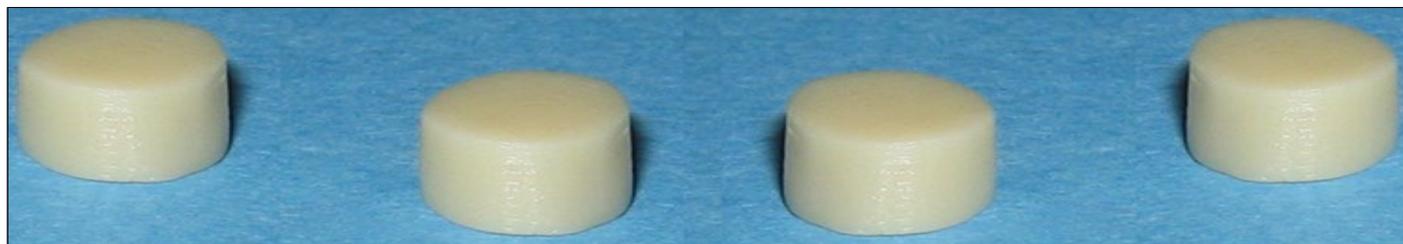


Figure 1. Zirconia discs.



Figure 2. Prepared tooth surface.



Figure 3. Cementation of Zirconia to tooth surface.

On comparatively evaluating the SBS between I_{A2} and II_{A2} a highly insignificant difference ($p=0.165$) was observed which shows that there was no difference between two Zirconia systems when bonded with Panavia F2 with thermocycling. On comparatively evaluating the SBS between I_{B2} and II_{B2} a highly insignificant difference ($p=0.961$) was observed which shows that there was no difference between two Zirconia systems when bonded with Rely X U200 with thermocycling.

On comparatively evaluating the SBS between I_{A1} and I_{A2} a highly significant difference ($p=0.004$) was observed which

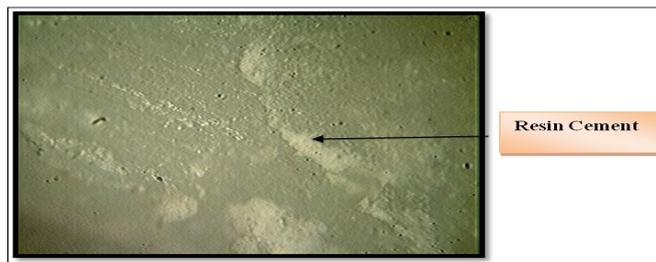


Figure 5. Cohesive failure as seen under stereomicroscope.

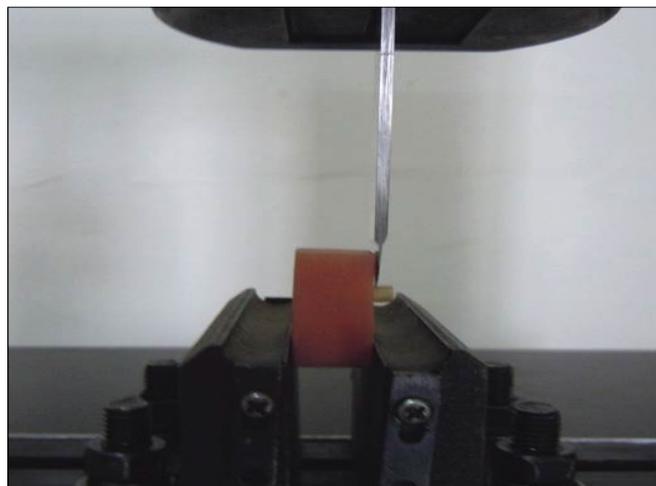


Figure 4. Sample loaded in Universal Testing Machine.

shows that thermocycling effects the shear bond strength of Cercon (Degudent, Hanau, Germany) when bonded with Panavia F2.O (Table 1).

On comparatively evaluating the shear bond strength between I_{B1} and I_{B2} a highly significant difference ($p=0.001$) was observed which shows that thermocycling effects the shear bond strength of Cercon (Degudent, Hanau, Germany) when bonded with RelyX U200 (Table 1).

On comparatively evaluating the shear bond strength between II_{A1} and II_{A2} a highly significant difference ($p=0.007$) was observed which shows that thermocycling effects the shear bond strength of Ziecon (Jyoti Lab Pvt Ltd, India) when bonded with Panavia F2.O (Table 2).

On comparatively evaluating the shear bond strength between II_{B1} and II_{B2} a highly significant difference ($p=0.005$) was observed which shows that thermocycling effects the shear bond strength of Ziecon (Jyoti Lab Pvt Ltd, India) when bonded with RelyX U200 (Table 2).

On comparatively evaluating the shear bond strength between I_{A1} and I_{B1} a highly significant difference ($p=0.047$) was observed which shows that Panavia F2.O has higher bond strength value than Rely XU200 when bonded with Cercon (Degudent, Hanau, Germany) after thermocycling (Table 3).

On comparatively evaluating the shear bond strength between I_{A2} and I_{B2} a highly significant difference ($p=0.038$) was observed which shows that Panavia F2.O has higher bond strength value than Rely XU200 when bonded with Cercon (Degudent, Hanau, Germany) without thermocycling (Table 3).

On comparatively evaluating the shear bond strength between II_{A1} and II_{B1} a highly significant difference ($p=0.049$) was observed which shows that Panavia F2.O has higher bond strength value than Rely XU200 when bonded with Ziecon (Jyoti Lab Pvt Ltd, India) after thermocycling (Table 4).

Table 1. Comparative evaluation of effect of Thermocycling on Shear Bond Strength of Cercon (Degudent, Hanau, Germany) when bonded with PanaviaF2.O and RelyX U200 with and without Thermocycling.

Groups	Mean	Std. Deviation	Std. Error Mean	95% Confidence Interval of the Difference		p value
				Lower	Upper	
I _{A1} * I _{A2} **	4.00143	2.29179	.86622	1.88187	6.12098	.004#
I _{B1} *** I _{B2} ****	3.71000	1.74940	.66121	2.09207	5.32793	.001#

#p value> 0.05 significant

I _{A1} * Shear bond strength of Zirconia Discs made of Cercon (Degudent, Hanau, Germany) bonded using PanaviaF2.O with thermocycling
I _{A2} ** Shear bond strength of Zirconia Discs made of Cercon (Degudent, Hanau, Germany) bonded using PanaviaF2.O without thermocycling
I _{B1} *** Shear bond strength of Zirconia Discs made of Cercon (Degudent, Hanau, Germany) bonded using RelyX U200 with thermocycling
I _{B2} **** Shear bond strength of Zirconia Discs made of Cercon (Degudent, Hanau, Germany) bonded using RelyX U200 without thermocycling

Table 2. Comparative evaluation of effect of Thermocycling on Shear Bond Strength of Zirconia Discs made of Ziecon (Jyoti Lab Pvt Ltd, India) when bonded with PanaviaF2.O and RelyX U200 with and without Thermocycling.

Groups	Mean Difference	Std. Deviation	95% Confidence Interval of the Difference		p value
			Lower	Upper	
II _{A1} * II _{A2} **	2.78429	1.82998	1.09184	4.47674	.007#
II _{B1} *** II _{B2} ****	2.50571	1.51845	1.10138	3.91005	.005#

#p value> 0.05 significant

II _{A1} * Shear bond strength of zirconia discs made of Ziecon (Jyoti Lab Pvt Ltd, India) bonded using PanaviaF2.O with thermocycling
II _{A2} ** Shear bond strength of zirconia discs made of Ziecon (Jyoti Lab Pvt Ltd, India) bonded using PanaviaF2.O without thermocycling
II _{B1} *** Shear bond strength of zirconia discs made of Ziecon (Jyoti Lab Pvt Ltd, India) bonded using RelyX U200 with thermocycling
II _{B2} **** Shear bond strength of zirconia discs made of Ziecon (Jyoti Lab Pvt Ltd, India) bonded using RelyX U200 without thermocycling

Table 3. Comparative evaluation of Shear Bond Strength of Cercon (Degudent, Hanau, Germany) when bonded with PanaviaF2.O and RelyX U200 with and without Thermocycling.

Groups	Mean Difference	Std. Deviation	95% Confidence Interval of the Difference		p value
			Lower	Upper	
I _{A1} * I _{B1} ***	1.35143	1.43411	.02509	2.67776	.047#
I _{A2} ** I _{B2} ****	1.64286	1.63829	.12769	3.15802	.038#

#p value> 0.05 significant

I _{A1} * Shear bond strength of zirconia discs made of Cercon (Degudent, Hanau, Germany) bonded using PanaviaF2.O with thermocycling
I _{A2} ** Shear bond strength of zirconia discs made of Cercon (Degudent, Hanau, Germany) bonded using PanaviaF2.O without thermocycling
I _{B1} *** Shear bond strength of zirconia discs made of Cercon (Degudent, Hanau, Germany) bonded using RelyX U200 with thermocycling
I _{B2} **** Shear bond strength of zirconia discs made of Cercon (Degudent, Hanau, Germany) bonded using RelyX U200 without thermocycling

On comparatively evaluating the shear bond strength between II_{A2} and II_{B2} a highly significant difference (p=0.037) was observed which shows that Panavia F2.O has higher bond strength value than Rely XU200 when bonded with Ziecon (Jyoti Lab Pvt Ltd, India) without thermocycling (Table 4).

When observed for nature of bond failure, results obtained showed that percentage of failure of Panavia F2.0 with Cercon before thermocycling were 14% Adhesive, 56% Cohesive, 28% Mixed and after thermocycling were 14%,72%,14% respectively and with Ziecon were 14%,42%,42% before thermocycling and 14%, 72% and 14% after thermocycling respectively. On the other side failure percentage of Rely X U200 with Cercon before were 28% Adhesive, 56% cohesive, 14% Mixed and after thermocycling 14%,72%,14% respectively and with Ziecon were 28%,42%,28% before

thermocycling and 28%,56%,14% after thermocycling respectively.

Discussion

All-ceramic dental restorations are metal-free alternatives to wide-spread metal-ceramic composite structures. Tetragonal stabilized zirconia (ZrO2-TZP) ceramic is the most recently introduced dental all ceramic material. It exhibits higher strength and toughness than all other commercially available dental ceramics and along with their translucency and brightness makes it is possible to achieve better esthetic results than with conventional metal-ceramic restorations [9-12].

Human dentin is a complicated bonding substrate due to its morphological and physical variations. Differing from the relatively homogenous and highly mineralized enamel, dentin

Table 4. Comparative evaluation of Shear Bond Strength of Ziecon (Jyoti Lab Pvt Ltd, India) when bonded with PanaviaF2.O and RelyX U200 with and without Thermocycling.

Groups	Mean Difference	Std. Deviation	95% Confidence Interval of the Difference		T	d.f.	p value
			Lower	Upper			
Π_{A1}^* , Π_{B1}^{***}	1.47000	1.57911	.00956	2.93044	2.463	6	.049 [#]
Π_{A2}^{**} , Π_{B2}^{****}	1.74857	1.73556	.14344	3.35370	2.666	6	.037 [#]

#p value > 0.05 significant

Π_{A1}^* Shear bond strength of zirconia discs made of Ziecon (Jyoti Lab Pvt Ltd, India) bonded using PanaviaF2.O with thermocycling
Π_{A2}^{**} Shear bond strength of zirconia discs made of Ziecon (Jyoti Lab Pvt Ltd, India) bonded using PanaviaF2.O without thermocycling
Π_{B1}^{***} Shear bond strength of zirconia discs made of Ziecon (Jyoti Lab Pvt Ltd, India) bonded using RelyX U200 with thermocycling
Π_{B2}^{****} Shear bond strength of zirconia discs made of Ziecon (Jyoti Lab Pvt Ltd, India) bonded using RelyX U200 without thermocycling

is heterogenous containing less mineral and more water and organic matrix which makes it challenging to achieve durable adhesive bonds [13].

The present study was undertaken to assess the SBS of two different Zirconia systems namely, Cercon and Ziecon bonded to dentin using resin cements Panavia F2.0 (Self etching) and RelyX U200 (Self adhesive) with and without thermocycling.

The variation in the bond strength observed could be due to variation in the chemical composition of the two cements used. RelyX U200 is composed of methacrylate monomers containing phosphoric acid groups which are all frequently used cross-linkers in adhesive systems. Panavia F2.0 on the other hand contains 10-methacryloxydecyl hydrogen phosphate which is a functional monomer. This variation in the functional monomer included in the adhesive solution and its particular molecular structure and affinity to hydroxyapatite plays a vital role in the adhesive performance of a resin luting agent.

The way molecules interact with hydroxyapatite-based tissues has been described in the Adhesion-Decalcification concept [14-16]. Molecules like 10-MDP from Panavia F2.0 chemically bond to Ca^{2+} of HAp forming stable calcium-phosphate and calcium-carboxylate salts, along with limited surface-decalcification. Panavia F2.0, with a pH of about 2.4 interacts superficially with enamel and dentin, and hardly dissolves HAp crystals, but rather keeps them in place, but complete resin penetration has been identified. Its monomers (HEMA, 10-MDP, 5-NMSA) [16], with low molecular weight, may have selectively diffused into dentin, forming the hybridized complex. The formation of an acid-base resistant zone in dentin has been recently reported adjacent to the hybrid layer in self-etch adhesive systems and may be postulated to influence the bond durability as it is more chemically and mechanically stable than normal dentin. On the other hand, RelyX U200 does not seem to contain monomers as capable of enhancing diffusion and lowering the initial viscosity of the mixture compared to Panavia. RelyX U200 contains multifunctional phosphoric acid methacrylates that are claimed to react with the hydroxyapatite of the hard tooth tissue when these monomers dissociate into methacrylate and the acidic phosphoric acid in an aqueous solution. It seems that the solvent was unable to generate enough interfibrillar spaces to accommodate the infiltrating adhesive. The results of this study are in accordance to the findings of the studies

done by ZohairyAA et al, Ernst CP and Wolfart M [17-19] that 10-MDP present in Panavia F2.0 is responsible for high shear bond strength values.

Thermal cycling was done for half of the samples of each group to evaluate the effect of changing intra oral conditions in mouth on the shear bond strength of Zirconia and dentin. In this study the samples were subjected to 5000 cycles with bath temperatures of 5^o-55^oC with a dwell time of 15 sec according to ISO standardization. According to Shahin, Kern and Blatz [20-21] the difference in the coefficient of thermal expansion between tooth structure and restorative materials might induce degradation of dentin/restoration surface.

Bond quality, however, should not be assessed on bond strength data alone, because the mode of failure is also important. This information may yield predictions of clinical performance. Following shear bond testing procedure, all the samples were observed under a Stereomicroscope at 80x magnification to identify the nature of bond failure, viz. cohesive, adhesive or a combination of both. Failure analysis revealed that failures were predominantly cohesive nature in the resin cement.

Monticelli et al and Eick in their studies [22,23] observed that resin tags generally broke off at the dentin surface rather than pulling out of the dentinal tubules suggesting that the bonding forces holding the resin tags to the tubule walls exceeded the cohesive strength of the resin tags. It can, thus, be stated that higher bond-strength values of the resin luting agent to both dentin and zirconia ceramic materials increases the cohesive failure rate within the adhesive cement. This finding is also in agreement with those of Altintas and Eldeniz [24] who in their study observed similar results.

This study revealed that the type and composition of Zirconia systems does not affect the shear bond strength of Zirconia to dentin. But on comparing the resin cements results showed a statically significant ($p > 0.05$) difference in their shear bond strength. Self-etching adhesive Panavia F2.0 performed better compared to self-adhesive resin cement RelyX U200. So, composition and type of resin cement affects the bond strength of Zirconia to dentin. Except these, it was also evaluated that the thermal cycling also affects the bond strength of both zircon systems to dentin with both adhesive cements. So, it shows that oral conditions also affect the bonding of Zirconia to dentin.

This in-vitro study also enabled us to assess the bond created by resin bonding agent between dentin and the

restorative material. However, in-vitro tests cannot adequately simulate clinical conditions in every detail. Subjecting the specimens to dynamic loading in artificial saliva before testing may closely resemble intraoral conditions with respect to hydrolytic degradation of the bond due to pH changes of saliva and the effect of temperature change in the mouth. Also, other clinically relevant factors such as configuration of cavity or crown preparation, dentin wetness, pulpal pressure, remaining dentin thickness and type of dentin (normal or sclerotic) should be considered when testing adhesive materials in-vitro. Even though bond strength is one of the parameters used more frequently to assess the quality of adhesive bond between

various substrates, there is no statistical correlation between that and other important characteristics like microleakage. Bonding performance may be predicted combining laboratory bond strength testing with microleakage/ nanoleakage tests, observation of marginal gaps and morphological analysis of bonding interface. The results of in vitro tests should, thus, be applied to the clinical situations with caution. It is admissible, however, to compare the measured in vitro results obtained under identical conditions. The final evaluation of material performance should be determined using long-term clinical studies which take the maximum number of parameters into account, least to mention, individual clinical determinants.

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