Shear bond strength of adhesive systems to enamel and dentin. Thermocycling influence

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Objectives: The purpose of the this study was to evaluate the influence of thermocycling on shear bond strength on bovine enamel and dentin surfaces of different adhesive systems. *Methods*: Thirty sound bovine incisors were sectioned in mesiodistal and inciso-cervical direction obtaining 60 incisal surfaces (enamel) and 60 cervical surfaces (dentin). Specimens were randomly assigned to 3 groups of equal size ($n = 40$), according to the adhesive system used: I—Single Bond; II—Prime & Bond NT/NRC; III—One Coat Bond. After 24-h storage in distilled water at 37^{o}C, each main group was divided into two subgroups: A-specimens tested after 24 h storage in distilled water at 37 °C; B - specimens submitted to thermocycling (500 cycles). Shear bond strength tests were performed. Data were submitted to ANOVA and Tukey test. *Results*: Means (MPa) of different groups were: I—AE-16.96, AD-17.46; BE-21.60, BD-12.79; II—AE-17.20, AD-11.93; BE-20.67, BD-13.94; III—AE-25.66, AD-17.53; BE-24.20, BD-19.38. *Significance*: Thermocycling did not influence significantly the shear bond strength of the tested adhesive systems; enamel was the dental substrate that showed larger adhesive strength; One Coat Bond system showed the best adhesive strength averages regardless of substrate or thermocycling.

1. Introduction

Since the introduction of acid conditioning, in 1955 by Buonocore [1], and of composite resins in 1962 by Bowen [2], extensive researches have been conducted to develop systems that bond effectively either to enamel as to dentin. The conditioning promotes central and peripheral dissolution of enamel prisms, demineralized peritubular and intertubular dentin, allowing that adhesive systems penetrate, polymerize and bond mechanically, minimizing marginal microleakage of salivary components and bacteria through the tooth/restoration interface, promoting increased longevity to the restoration [3].

One of the biggest concerns of Restorative Dentistry is to find a material that, apart from recovering the dental function, presents mechanical properties similar to dental structure, good marginal adaptation and is biocompatible, besides of reproducing the natural tooth color and preserving the most sound structure.

There is a great number of commercially available adhesive systems nowadays, which can confound the professional when choosing the adequate material for different clinical situations [4]. Adhesion to dentin is more complex than to enamel, due to its structural features and organic contents [5]. To obtain an optimized adhesion to dentin of the most recent adhesive systems, it is required knowledge of the smear layer, its influence on substrates and adhesion, and how this layer is to be conditioned before restorative procedure [6].

In this way, the purpose of the present study was to evaluate the influence of thermocycling on shear bond strength of different modern adhesive systems when applied on bovine enamel and dentin surfaces. The null hypothesis was that there is no influence of the the thermocycling on the shear bond strength of adhesives systems, regardless of the substrate.

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TABLE I Specifications of tested materials

Adhesive System	Composition	Manufacturer	Batch
Single Bond (SB)	Total-etch, 2 steps/ 35%PA, BIs-GMA HEMA/ water ethanol, polyacrylic polyitaconic copolymer	3M/ESPE Dental Products St Paul MN 55144 - USA	9DC
Prime & Bond NT/NRC (PB)	Self-etch, 2 steps/ NRC: 30% maleic acid, itaconic acid, methacrylate monomers; Adhesive: PENTA, UDMA, R5-62-1, T-resin, D-resin, nano-fillers initiators, cetylaminehydrofluoride, stabilizer, acetone	Dentsply De Trey GmbH De-Trey – Str – 1 D – 78467 Konstanz Germany	NRC: 0105001250 Adhesive: 0106000536
One Coat Bond (OC)	Total-etch, 2 steps/ 15% phosphoric acid gel, HEMA, HPMA, MMA, UDMA/ water base	Coltène/Whaledent Inc. 750 Corporate Drive Mahwah, NJ 07430/USA	ID 164

2. Materials and methods

Thirty sound bovine incisors, extracted within a six-month period and stored in azide solution at 4 °C were selected and cleaned with periodontal scaler and water/pumice slurry in dental prophylactic cups. Roots were sectioned 2 mm below the amelocemental junction and then, according to the adhesive system used (Table I), teeth were randomly assigned to 3 groups of equal size. Each tooth was sectioned in mesiodistal and inciso-cervical direction with a water-cooled diamond saw in a sectioning machine (Minitom, Struers, A/S, Copenhagen, DK-2610, Denmark), obtaining 60 incisal surfaces (enamel) and 60 cervical surfaces (dentin).

Surfaces were identified and individually embedded in polyester resin using PVC cylinders (2.1 cm diameter; 1.1 cm high). After polymerization, specimens were grounded under water refrigeration in a polishing machine (Politriz, Struers A/S, Copenhagen, DK-2610, Denmark) using #120 and #400—grit silicon carbide paper to superficial (incisal) or overlying enamel and to expose a flattened, smooth middle dentin surface (cervical). Complementary grounding was accomplished with #600 SiC paper for 30 s to produce and standardize the smear layer.

To delimit the substrate bonding site, a small piece of insulating tape with a central hole was attached to specimen's surface. The tape perforation was made by means of a modified Ainsworth rubber-dam punch machine to provide 3 mm diameter holes. The limitation of bonding area has a three aim: to define a fixed test surface, so that calculations of bonding strength will be related solely to the evaluated area; to avoid excess of adhesive on dentin surface, which could compromise the distribution of tensions during the test and hence the validity of results; to ensure that the restorative material would be further correctly placed thus forming the resin cylinder exactly on the desired site, since it is difficult to visualize the area to be evaluated, once the matrix is placed.

Specimens were randomly assigned to 3 groups of equal size ($n = 40$), according to the adhesive system used: I—Single Bond; II—Prime & Bond NT/NRC; III—One Coat Bond. The tested materials with their compositions, specifications and manufacturers are listed in Table I.

The tested adhesive systems were applied on enamel and dentin surface, strictly following the manufacturer's instructions. For Group I, Single Bond/3M (ethanol/water based, single-bottle bonding agent) was used. The surface was treated with 37% phosphoric acid gel (Total Etch, Vivadent Ets., FL-9494, Schaan, Liechtenstein) for 10 s, rinsed for 10 s, gently dried, and then the adhesive applied in two consecutive layers; the remaining solvent was evaporated with a brief, gentle dry air jet and was light-cured for 20 s (XL 3000, 3M Dental Products, St Paul, MN 55144 with a 450 mW/cm^2 output).

For Group II, Prime & Bond NT/NRC (self-etching primer adhesive system) was used. Surfaces were conditioned with NRC (Non-rinse Conditioner, Dentsply DeTrey Konstanz, Germany, D-78467) left for 20 s and the excess of primer was removed and Prime & Bond NT was applied on all surface, left 20 s, the excess solvent was removed with dry air jet for 5 s, resulting a surface with a glazed and uniform appearance; photopolymerized for 20 s.

For Group III, One Coat Bond/Coltène (water based, one-bottle bonding agent) was used. The surfaces were conditioned with 15% phosphoric acid gel (Etchant 15, Coltène/Whaledent Inc., Mahwah, NJ07430, USA) for 30 s; rinsed thoroughly for 20 s, gently dried with absorbing paper to remove water excess and keep tooth surface moist and the bonding system was applied to etched surface with a light scrubbing motion for 20 s, air-dried for 2 s and light-cured for 30 s.

After bonding procedure, each specimen was fixed in a clamping metallic device (developed by Houston Biomaterial Research) in such way that the substrate site remained parallel to a flat surface. A split bisected Teflon matrix was positioned over the tooth/resin block resulting in a cylindrical cavity with a 3 mm diameter and 4 mm high.

A hybrid light-cured composite resin (Filtek Z250) was inserted into the matrix in three increments, each one polymerized for 40 s. As the matrix cavity was completely filled, the specimen was removed from the clamping device, the matrix was opened and separated, leaving adhered to the delimited dentin surface a composite resin cylinder with a 3 mm diameter and 4 mm high.

After 24-h storage in distilled water at 37 °C, each main group was divided into two subgroups ($n = 20$) according to thermocycling: T—specimens submitted to a thermocycling regimen of 500 cycles between 4 °C and 55 °C water baths after 24-h storage. Dwell time was 1 min, and there was a 3-s transfer time between baths; WT—specimens were tested after 24 h storage in distilled water at 37 °C. Shear bond strength tests were made using a Universal Testing Machine, at a crosshead speed of 0.5 mm/min and a 50 kgf load cell until fracture. Bond strength values were calculated in kgf/cm^2 and translated into MPa.

Data were analyzed as to their distribution and as normal distribution was observed. In such case, data could be analyzed by two-way ANOVA and for individual comparison and Tukey test could be applied with a 0.05 significance level.

Bonding failure sites were not analyzed statistically. Enamel and dentin bonding areas were viewed under a stereoscopic optical magnifier (40×) to assess the type of failure: *adhesive* failure was considered to be the one at the specimen/adhesive interface; *cohesive* failure occurred in the material or the substrate, with no damage to the interface and finally; *mixed* failure was the one involving at the same time the interface and the material.

Scanning electron microscopy of the interface of each adhesive system with the substrate was made. Specimens were sectioned at a 90° angle to the bonding interface and manually smoothened with #1000- to #4000-grit SiC paper. The dentin/adhesive interface was conditioned with a 37% phosphoric acid gel for 5 s, thoroughly rinsed with distilled water and immediately immersed in 2.5% glutaraldehyde in 0.1 M cacodylate buffer, pH 7.4, for 12 h at 4 °C. After fixing, the samples were washed with cacodylate buffer several times, sequentially dehydrated in an alcoholic series and then immersed in 100% hexamethyldisizilane for 10 min and dried in an exhaust system.

Specimens were mounted on stubs with their treated surfaces face up, using cooper adhesive and sputter-coated with gold. The adhesive/dentin interfaces were examined with a JSM T330A scanning electron microscope (Jeol Ltd., Tokyo 190-0012, Japan) operating at 20 kV. A standardized series of photomicrographs were taken at different magnifications. Two previously calibrated examiners analyzed the interfaces independently. For all the adhesive systems were analyzed and the thickness of the hybrid layer was measured solely in those areas considered representative of the sample. For each measurement, a consensus was always reached between the examiners.

3. Results

Table II show the means of different gruops as related to the dental substrates and adhesive systems, whether thermocycled or not.

Data analysis disclosed that thermocycling did not influence shear bond strength of tested adhesive systems, regardless of system or substrate.

As related to adhesive systems, One Coat Bond presented the best average (21.9 MPa) and statistical difference in compariosn with other adhesive systems (Prime & Bond NT/NRC and Single Bond), which exhibited statistical similarity ($p < 0.05$) between them (15.93 and 17.88 MPa respectively).

As to substrates, it was noticed that enamel (21.05 MPa) showed statistically significant difference in relation to dentin (15.92 MPa).

As to the interaction among adhesive systems and thermocycling, it was observed that this treatment did not affect the systems' behavior. However, comparative analysis of adhesive × thermocycling × substrate, revealed that only Single Bond presented statistical difference between enamel and dentin, and the last one presented the lowest value.

As substrates were related to adhesives, it was observed that One Coat Bond showed high value in enamel and dentin, and Prime & Bond NT/NRC showed the lowest strength averages for both substrates.

Failure after testing occurred mostly involving at the same time the bonding interface and the adhesive system/composite resin (*mixed* failure) as may be seen on Figs. 1 and 2.

Scanning electron microscopy of the polished cross sections of the bonded specimens presented for *ONE COAT BOND*—In bovine enamel, this adhesive showed a thin hybrid layer with resin tags formation in demineralized areas due to etching. Its performance was similar in Control Group as in the thermocycled one. In bovine dentin, there was a thin hybrid layer (□2.67 □m) in all dentin/resin interfaces. Many resin tags were observed next to the conditioned area, as well as lateral branches. There was gap formation (□4 □m) in some interface areas of the analyzed Control Group, which has not occurred with thermocycled Group. (Fig. 3(A) and (B)).

SINGLE BOND—In bovine enamel, this adhesive presented resin tags larger than One Coat Bond. This was the most representative behavior to both Groups. In bovine dentin, a thick hybrid layer (approx. 3.4 □m) was observed with resin tags, a thick hybrid layer (approx. 3.4 □m) was observed next to conditioned area, though in lower proportion. The behavior of both Groups (Control and Thermocycled) was similar. (Fig. 4(A) and (B)).

PRIME & BOND NT/NRC—In bovine enamel, there was a thin hybrid layer with few resin tags, which were absent in some areas. In bovine dentin, the SEM showed a thin hybrid layer, but with many resin tags. (Fig. 5(A) and (B)).

TABLE II Means (MPa) and standard deviations of the different groups studied

	Enamel		Dentin	
	WT	T	WT	T
Single Bond	16.96(5.84)bcd	21.60(5.15)ab	17.46(2.42)abc	12.79(6.91)cd
Prime & Bond NT/NRC	17.20 (2.88)bcd	20.67(7.09)ab	11.93(4.62)d	13.94(6.19)cd
One Coat Bond	25.66(5.63)a	24.20(6.87)a	17.53(6.15)abc	19.38(7.82)abc

Same letters indicate statistical similarity.

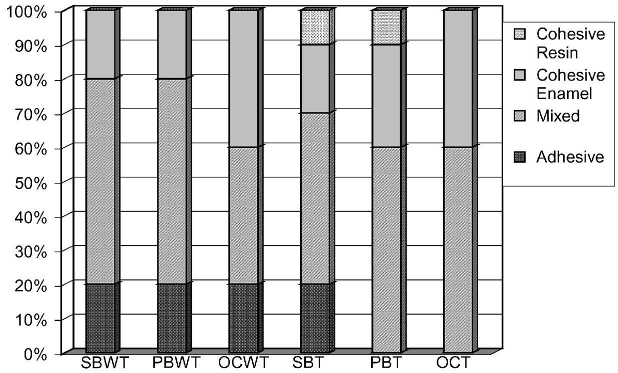


Figure 1 Types of failure in enamel to different groups studied.

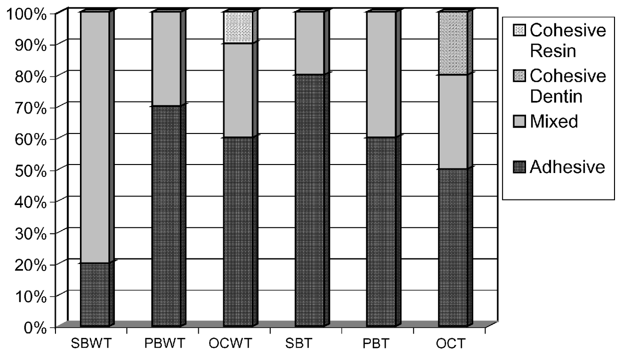
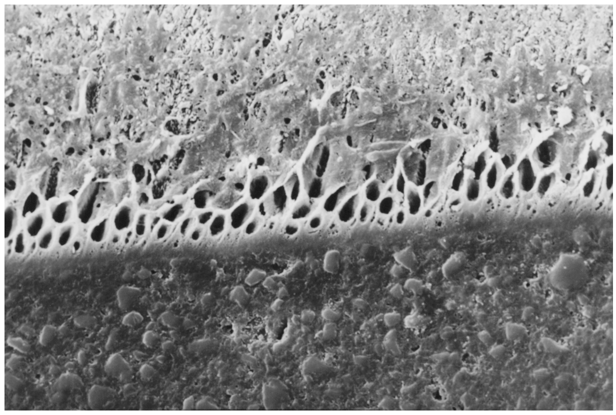
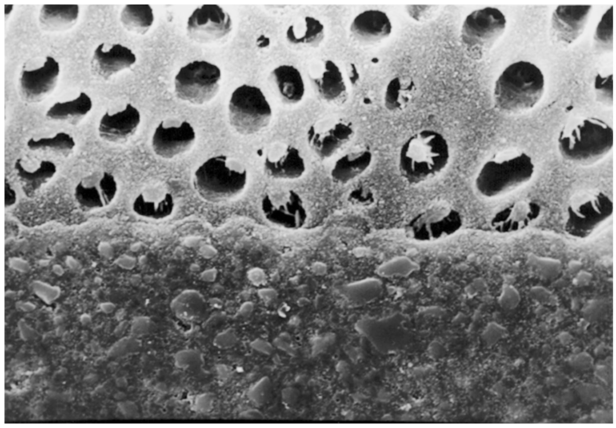


Figure 2 Types of failure in dentin to different groups studied.



(A)



(B)

Figure 3 (A) SEM of the resin-enamel interface of a polished cross section of bovine enamel etched with 35% phosphoric acid (PA), rinsed and bonded with One Coat Bond. The hybrid layer (H) is thin and there are long resin tags (T) (magnification ×1500). (B) SEM of the resin-dentin interface of a polished cross section of bovine dentin etched with 35% phosphoric acid (PA), rinsed and bonded with One Coat Bond. The hybrid layer (H) is thin and there are resin tags (T); the superficial lateral branches are filled with resin. (magnification ×1500).

4. Discussion

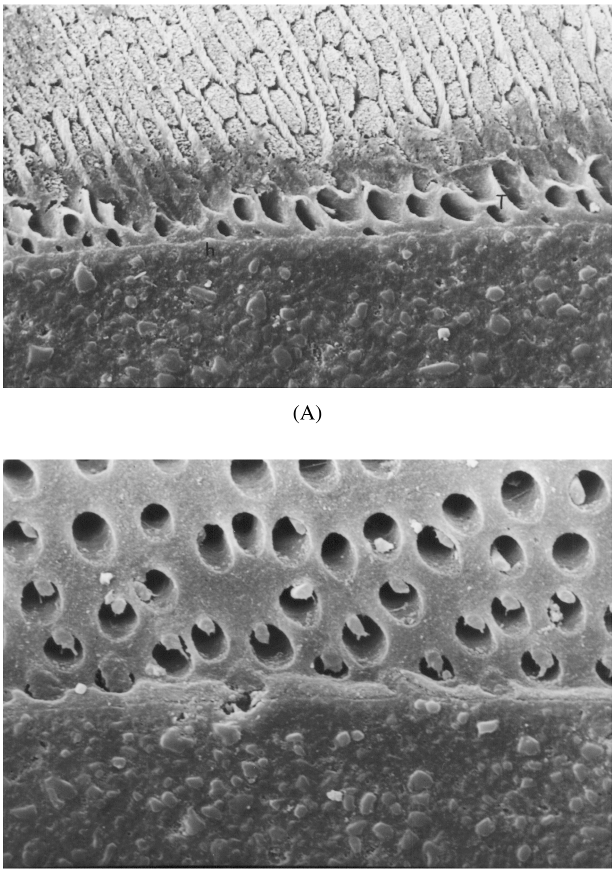
In the present study, it was observed that thermocycling did not influence the shear bond strength of tested adhesive systems, regardless of dental substrate employed. A suitable explanation to this fact is the evolution of adhesive systems composition, which, nowadays, seem to have better chemical stability, which might result in a greater longevity.

Although according to Martuci *et al.* [5], is very dificult correlation studies *in vitro* with *in vivo*, when used the thermocycling to verify this influence on restorative materials, but this influence its is a parameters employed in different works [7, 8]. Other authors [7, 8] reported that thermocycling might even increase, decrease or even have no influence on adhesion. This result was divergent from the study that reported that teeth storage medium before testing could reduce the bond strength to dentin, and this adhesion was influenced by storage duration and thermocycling [9]. Although bonding strength tests are not accurate to characterize adhesive systems effectiveness, they are a good way to compare different adhesive systems following the same parameters [10]. In this way, they are important to validate restorative materials.

Among the adhesives tested in the present study, One Coat Bond showed the best shear bond strength, followed by Single Bond and Prime & Bond NT/NRC. some studies demonstrated that adhesive systems with load particles show lower bonding strength to enamel [11, 12, 13]. This fact seems to be related to high viscosity of these adhesive systems, which are unable to penetrate in de-mineralized areas as deeply as adhesives without load [14]. This might explain the fact of Prime & Bond NT exhibit the lowest bonding value in the present study, as compared to the other tested systems, because this system include load particles in its formulation, which hinder the penetration in all de-mineralized areas, as revealed by the SEM study of interface.

In relation to adhesion to different dental substrates, independently of the adhesive system applied, it was observed that the best results were in enamel, due to the fact that adhesion to dentin is more difficult than to enamel because of dentin's morphologic features, such as high organic content and tubular structure with odontoblastic process, these results might be expected. The SEM confirms it, revealing that enamel showed the best results of bonding strength produced by a better interaction to this substrate as compared to dentin. Apart from this, the new adhesive systems include low molecular weight hydrophilic monomer in their composition and use acetone/ethanol as vehicle, improving their wetting and penetration capacity on enamel surface, thereby increasing its adhesion [15, 16].

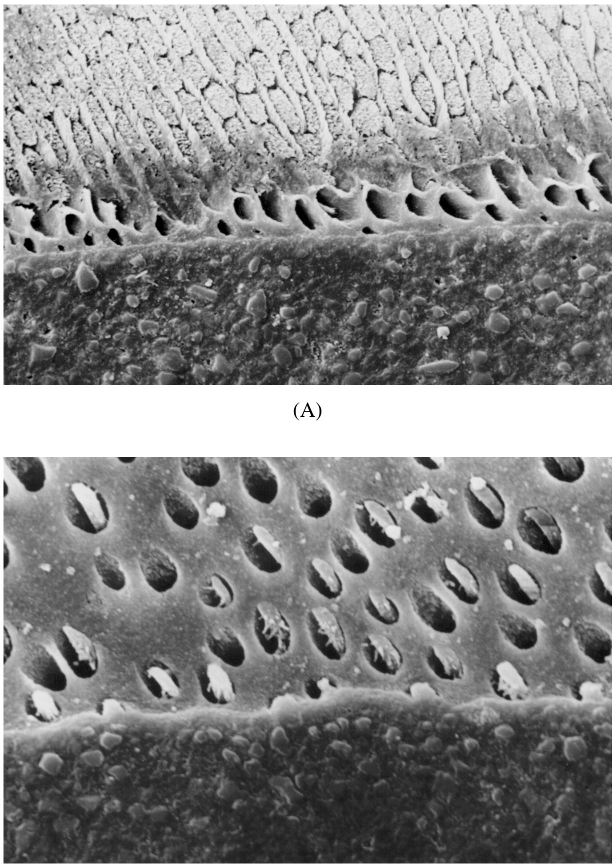
According to Finger and Fritz [17], high shear bond strength values are obtained by the use of one-bottle bonding systems. This may be related to the acetone



(A)

(B)

Figure 4 (A) SEM of the resin-enamel interface of a polished cross section of bovine enamel etched with 34% phosphoric acid (PA), rinsed and bonded with Single Bond. The hybrid layer (H) is thin and with resin (T). (magnification ×1500). (B) SEM of the resin-dentin interface of a polished cross section of bovine dentin etched with 35% phosphoric acid (PA), rinsed and bonded with Single Bond. The thick hybrid layer (H), funnel-shaped resin tags (T), and their lateral branches can be seen. (magnification ×1500).



(A)

(B)

Figure 5 (A) SEM of the resin-enamel interface of a polished cross section of bovine enamel etched with NRC (15% phosphoric acid -PA) and bonded with Prime & Bond NT. There is a hybrid layer (H), but no resin tags. (magnification ×1500). (B) SEM of the resin-dentin interface of a polished cross section of bovine dentin etched with NRC (35% phosphoric acid -PA) and bonded with Prime & Bond NT. Very thin hybrid layer (H) and many resin tags (T), and their lateral branches filled with resin. (magnification ×1500).

solvent included in their composition. The water-removing capacity of acetone induces a more complete wetting and resin penetration into the etched substrate. Acetone and ethanol are volatile substances that could easily evaporate from bottles during use of adhesive systems [18]. Its presence in some adhesive compositions is crucial for the penetration of this material on moist dentin, and the evaporation of the solvent in over 3 weeks of simulated use may decrease its concentration, therefore decreasing the reactivity of the adhesive on moist dentin [19]. This might explain the fact why Prime & Bond NT, which is an acetone-based system, showed this behavior in the present study. For Single Bond, the presence of water in its composition might be beneficial because water is able to reopen the collapsed network of collagen fibers on dry spots inadvertently left on the surface and prevent the formation of "ghost" hybrid layers [20, 21].

Even though some authors conclude that the thickness of hybrid layer is proportional to union strength of adhesive systems in dentin [22], in the present study, by SEM observations, it was observed that One Coat Bond presented smaller hybrid layer than Single Bond, despite its bonding strength being better. As regards enamel, adhesion depends on the amount of resin tags formed, so the analysis of resin tags formed on interface observed by SEM, can explain why the adhesion of One Coat Bond is more significant than the other systems' in the conducted study.

A feature that hampers the comparison to other studies in the existing literature is the use of bovine teeth which, according to LOPES *et al.* [23], may alter the results, due to its different structure as compared to human teeth.

Although bonding strength tests are not accurate, in order to characterize adhesive systems effectiveness, these tests constitute a good way to compare, following the same parameters, different adhesive systems [10]. In this way, adhesives are important to validate restorative materials.

5. Conclusion

Based on the findings of this research, the null hypothesis was supported. The thermocycling did not influence significantly the shear bond strength of the tested adhesive systems. Enamel was the dental substrate that showed larger adhesive strength and the One Coat Bond system showed the best performance regardless of substrate or thermocycling.

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