

ORIGINAL ARTICLE

Effects of thermomechanical and thermocycling on the shear bond strength of brackets to bovine enamel bonded with conventional and self-etching adhesive systems

Efeito da ciclagem térmica e térmico-mecânica na resistência adesiva ao cisalhamento de bráquetes aderidos ao esmalte bovino com sistema adesivo convencional e auto-condicionante

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ABSTRACT

Several adhesive systems have been developed in the last decade. The aim of the current study was to evaluate the shear bond strength (SBS) of orthodontic brackets bonded with conventional primers (XT) or self-etching primers (SEP), by means of thermocycling (TC) and thermomechanical (TM) procedures. 90 bovine incisors were randomly divided into 2 groups (45 specimens each) for brackets bonding: XT (Transbond XT, 3M-Unitek, CA, USA); SEP (Transbond Plus SEP, 3M-Unitek, CA, USA). After bonding, groups were divided into 3 subgroups: NC (no cycling)- 1 and 4 (n = 15 each), TMC - 2 and 5 (n = 15 each); and TC- 3 and 6 (n = 15 each). The fatigue simulator was used for the thermal fatigue test of groups 3 and 6, which were submitted to thermal variation cycles - 500 cycles were carried out in between 5 and 55 C (ISO 11405). Groups 2 and 5 were submitted to thermomechanical tests by using the mechanical fatigue simulator. The specimens were placed at a metal base at an angle of 45, in a way that the 2.5 mm diameter metal needle was fixed in the upper part of the cycling machine and could induce impulses of 1 bar load intensity and 4 Hz frequency (4 cycles per second) on the bracket. The samples were submitted to 100 000 mechanical cycles and 500 thermal cycles which varied from 5C to 55C. The SBS test was performed in a universal testing machine (1 mm/min). After debonding, optic microscopic evaluation for adhesive penetration and Adhesive Remaining Index (ARI) analyses in the enamel surface were performed. The results showed higher SBS (14.70 ± 4.85 MPa) values for SEP. The SBS remained similar despite cycling (NC-11.44 MPa; TC-11.20 MPa; TMC-11.19 MPa) for XT, meanwhile for SEP, the TMC subgroup

RESUMO

Muitos sistemas adesivos foram desenvolvidos na última década. O objetivo desse estudo foi avaliar a resistência adesiva ao cisalhamento (RAC) de bráquetes ortodônticos aderidos com primer convencional (PC) ou primer auto-condicionante (PAC), submetidos à ciclagem térmica (CT) e ciclagem térmico-mecânica (CTM). Incisivos bovinos (n = 90) foram randomicamente divididos em 2 grupos (n = 45) para a colagem dos bráquetes: XT (Transbond XT, 3M-Unitek, CA, USA); SEP (Transbond Plus SEP, 3M-Unitek, CA, USA). Após o procedimento adesivo os grupos foram subdivididos em 3 sub-grupos: SC (sem ciclagem) - 1 e 4 (n = 15 cada); CTM (ciclagem térmico-mecânica) - 2 e 5 (n = 15 cada); CT (ciclagem térmica) - 3 e 6 (n = 15 cada). Um simulador de fadiga térmica foi usado para os subgrupos 3 e 6, que foram submetidos a 500 ciclos térmicos entre 5 and 55 C (ISO 11405). Os subgrupos 2 e 5 foram submetidos à ciclagem térmico-mecânica em um simulador de fadiga. Os espécimes foram colocados em uma base de metal num ângulo de 45 e os braquetes receberam impulsos de 1 bar e 4 Hz de frequência, por uma agulha de metal de 2.5 mm de diâmetro fixada na haste superior do equipamento. Foram submetidos a 100.000 ciclos mecânicos e 500 ciclos térmicos entre 5 and 55 C. O teste de RAC foi realizado em uma máquina universal de testes (1mm/min). Após o rompimento da união foi realizada uma avaliação em microscopia óptica para avaliar a penetração adesiva e o Índice de Permanência do Adesivo (IPA) sobre o esmalte. Os resultados mostraram maiores valores de RAC ($14,70 \pm 4,85$ MPa) para o SEP. A CT não influenciou os resultados para o XT (NC- 11,44 MPa; TC - 11,20 MPa; TMC - 11,19 MPa), enquanto para o SEP, a CTM mostrou maiores valores (16,84 MPa). O

showed higher values (16.84 MPa). 2-way ANOVA test indicated no statistical significant interaction effect for cycling. The primer effect (XT x SEP) was statistically significant. Enamel ARI values were higher for SEP/TMC (ARI=3). The results showed that SEP is promising in orthodontic practice due to adequate values of SBS, even under TC or TM fatigue.

KEYWORDS

Shear bond strength; Thermal cycling; Mechanical cycling

teste ANOVA 2 fatores não evidenciou significância estatística para a interação com a ciclagem. O fator primer (XT x SEP) foi estatisticamente significativo. O IPA foi maior para a interação SEP/CTM (IPA = 3). Os resultados evidenciaram que o SEP é promissor para uso na prática ortodôntica devido aos valores de RAC sob condições de ciclagem térmica e mecânica.

PALAVRAS-CHAVE

Resistência adesiva ao cisalhamento; Ciclagem térmica; Ciclagem mecânica.

INTRODUCTION

One of the main challenges for modern clinicians is to obtain a good adhesion of materials to dental structure, once it is necessary for both preventive and restorative procedures.

Since 1955, when Buonocore introduced the acid-etching bonding technique, the concept of bonding resin-based materials to enamel has favoured the development of its application in preventive and restorative procedures in dentistry, including the bonding of orthodontic brackets [1]. From the time when it became available, phosphoric acid has been considered to be the best choice for total acid etching. In 1974, Moin and Dorgan investigated the effects of different concentrations of phosphoric acid on surface enamel, and found that the most consistent and adequate concentrations to etch enamel in preparation for bonding ranged from 30% to 40%. The enamel surface layer, which can be modified by acid etching, may vary from 10 μm to 30 μm , while the cleaning process after adhesive removal can remove until 55.6 μm of the enamel surface [2].

Several adhesive systems have been developed in the last decade. These materials can be divided in two groups according to their clinical applications. The first group includes enamel etching with phosphoric acid, followed by the application of primer/resin adhesive.

The second group includes one self-etching primer, which combines both acid etching with the primer action in one single step, followed by the application of resin adhesive. Although the most effective etching step considered occurs when using phosphoric acid, there still have some disadvantages for surface enamel after the debonding of orthodontic brackets. On the contrary, self-etching primers have shown a limited etching pattern, due to its relatively greater pH compared to phosphoric acids, and therefore less likely to cause damage to dental enamel [3].

In order to minimize the number of steps when bonding and to reduce clinical chair time, this new group of adhesives were introduced in the market and called "self-etching adhesives" [4]. They combine conditioning and priming into a single treatment step, and do not require acid etching and rinsing as for the conventional union agents. An important feature of the self-etch approach is that infiltration of resin occurs simultaneously with the self-etching process [5].

Brackets are also subjected to compressive forces [1]. Bond strength is influenced by various factors such as the surface area, conditioning procedures, type of adhesive used, bracket base design, the treatment of the bracket base and protocol followed during bonding. Ideally, brackets bonding must be able to resist mastication during service and should

be easily removed at the end of the treatment with minimal damage to dental surface [6]. However, the majority of the in vitro studies have not used any type of artificial load previous to the bonding test, and therefore, some authors recommend thermocycling and/or thermo mechanical cycling, in order to consider the real longevity of bonding [7].

Several studies have been performed to evaluate how to reduce enamel damages caused by debonding procedures. Hence, in order to investigate adhesive properties of materials used for orthodontic bonding, it is important to evaluate its performance regarding the stress involved in the bracket-adhesive-enamel system [8]. For that matter, the aim of the current study was to evaluate SBS of orthodontic brackets bonded with conventional primers or self-etching primers, artificially aged by means of thermocycling and thermomechanical procedures.

MATERIAL AND METHODS

Sample

Ninety bovine incisors were used in the investigation. The roots were sectioned in the amelo-cementary junction. All teeth were embedded in PVC tubes with acrylic resin, and the vestibular face was positioned facing the base. The specimens were polished on a laboratory polishing wheel using 200, 400 and 600-grit water sandpapers (3M, Brazil). The final dimensions were 2.5 cm diameter and 2.5 cm height and the vestibular face showed a minimum 25 mm² area of exposed enamel.

All samples were randomly divided in 2 groups (n = 45), according to the primer used for bracket bonding. XT Group—hydrophobic conventional primer (Transbond XT, 3M-Unitek, CA, USA); SEP Group—self-etching primer (Transbond Plus SEP, 3M-Unitek, CA, USA). All specimens were bonded with XT Transbond resin adhesive (3M-Unitek, CA, USA).

Bracket Bonding

After cleaning, the surfaces were washed with distilled water for 10 s and dried with air

spray. The orthodontic metal brackets were bonded according to the adhesive material and following the manufacturer's instruction. The photoactivation process was performed by means of a LED device (Schuster Emitter, Schuster Comércio de Equip. Odontont. Ltda, series number 140 uLX, light power: 1250 mW/cm², wavelength: 420 to 480nm, frequency: 50/60 Hz, multi-voltage).

. Following that, each group was subdivided in 3 groups (n =15): NC Group (no cycling) – a shearing test was performed with no previous cycling. TMC Group (thermo mechanical cycling) – thermomechanical cycling of the samples previous to the SBS test. TC Group (thermocycling) – the samples were submitted to thermal cycles before the SBS test.

For groups 1, 2 and 3, the surface was etched with 37% phosphoric acid (3M, ESPE, USA) for 30 seconds, washed with distilled water and the surface was dried with air . The XT Transbond primer was applied on the surface and it was further slightly dried. The bracket was positioned and firmly bonded with XT Transbond adhesive in its base, the excess was removed and a 10 s polymerization was performed along each face (mesial, distal, cervical and incisal) of the bracket. For groups 4, 5 and 6, the active etching was performed by means of the application of the Transbond Plus Self Etch Primer SEP (3M, Unitech Miami/USA), both acid and primer incorporated, scrubbing the surface of the enamel for 5 s with its own dispenser and following the manufacturer's instructions. The surfaces were slightly dried and the metallic brackets for central incisors were bonded with XT Transbond resinous adhesive and photopolymerized for 10 s in each face.

Groups 1 and 4 were not submitted to any type of cycling, and the shear bond test was performed immediately after the bonding of the brackets.

The fatigue simulator (ER 11000, ERIOS, São Paulo, SP, Brazil) was used for the thermal fatigue test of groups 3 and 6, which were submitted to thermal variation cycles - 500

cycles were carried out in between 5 °C and 55 °C (ISO 11405) [9].

Groups 2 and 5 were submitted to thermomechanical tests by using the mechanical fatigue simulator (ER 11000, ERIOS, São Paulo, SP, Brazil). They were placed at a metal base at an angle of 45°, in a way that the 2.5 mm diameter metal needle was fixed in the upper part of the cycling machine and could induce impulses of 1 bar load intensity and 4 Hz frequency (4 cycles per second) on the bracket. The samples were submitted to 100 000 mechanical cycles and 500 thermal cycles which varied from 5 °C to 55 °C.

SBS resistance test

A Universal Testing Machine EMIC DL2000 (EMIC Equip. Sist. Ensaio Ltda., São José dos Pinhais, PR, Brazil) was used for the collection of the values of SBS resistance. The values were measured at a crosshead speed of 1 mm/min and a 100-kg load cell.

The required load for the debonding process or to start the fracture was registered in kilogram force (kgf) per millimeter squared (mm²) by means of a computer connected to the EMIC, and the values obtained were based on the base surface of the bracket and then converted into megapascals (MPa).

Adhesive Remnant Index (ARI)

After debonding, teeth and brackets were collected, and photomicrographs were prepared in a stereomicroscope (ZEIS Stemi 200 - C) connected to a digital camera (Sony Cybershot 4.1 megapixels), to determine the amount of

adhesive remaining on tooth surfaces. The ARI score used in this investigation was the same proposed by Artun and Bergland [10], in 1984. ARI ranged from 0 to 3, following the scores defined as follows: 0 = no adhesive left on the tooth; 1 = less than half of the adhesive left on the tooth; 2 = more than half of the adhesive left on the tooth; 3 = adhesive totally left on the tooth with a distinct impression of the bracket mesh.

Scanning Electron Microscopy (SEM) - analysis

In order to evaluate the amount of adhesive remaining and adhesive penetration on the enamel surfaces, two specimens from each group were submitted to SEM after the SBS resistance test. For that matter, each specimen was sectioned in a way that only the enamel surface containing the bracket was used for the microscopic analysis. The specimen section (6 mm diameter) was performed by means of a MicroMill machine (Manrod Quality Machines), and then placed in a sputter coater (Denton Vaccum – Desk II). The specimens were examined by means of image capture, (figures 1 and 2) through a specific software connected to the Scanning Electron Microscope (JSM - 840^a Geol, Tokyo – Japan), of 20 kV energy.

RESULTS

The statistical analysis on SBS for the experimental groups are presented in Tables 2,3,4 and 5 below.

Table 1 - Material composition

	Brackets	Transbond XT Adhesive	Transbond Plus Self Etch Primer
Composition	17-4 steel 80-micron base mesh	BisGMA, silano, n- dimetylbenzocaine and hexa-fluoride-phosphate	Mono - and di-HEMA phosphates, canphoroquinone, distilled water, aminobenzoate, Potassium Hexafluorotitanate, Butylhydroxytoluen, methylparaben e propylparaben
Manufacturer	3M Unitek USA	3M Unitek, USA	3M Unitek USA

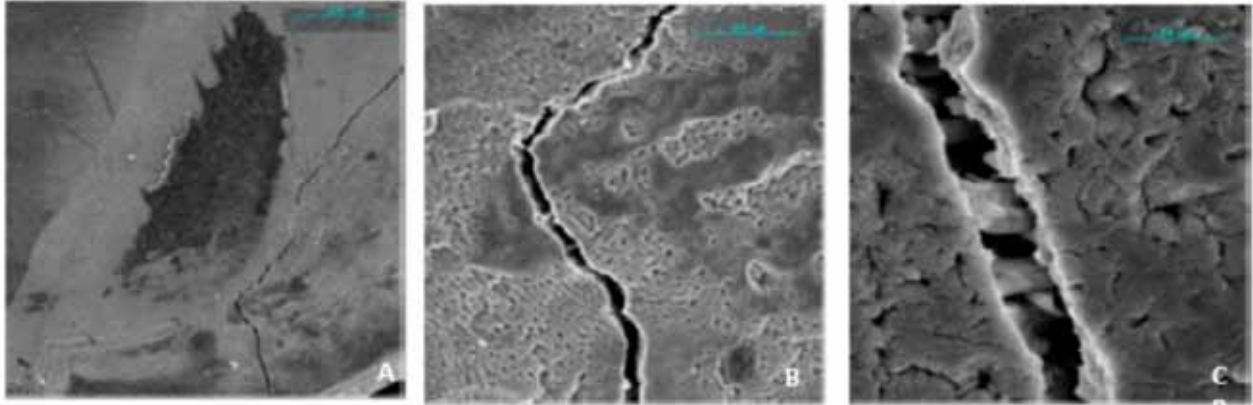


Figure 1 - XT/TC groups micro image: A) 100x – enamel cracking; B) 500x; C) 2000x – cracking detail.

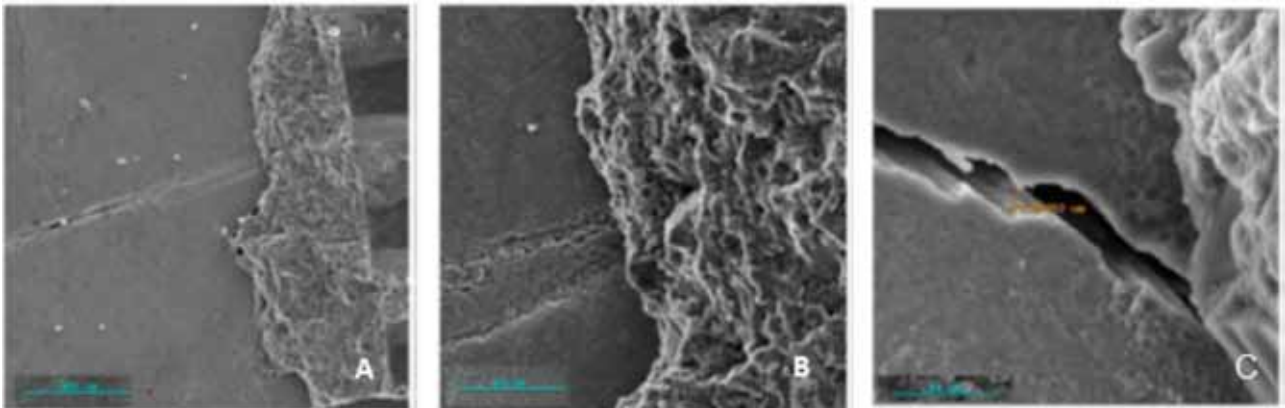


Figure 2 - Micro images of SEP/NC: A) 100x – amount of enamel cracking; B) 500x; C) 2000x cracking detail.

Table 2 - Descriptive statistics in MPa - Mean values (\pm standard deviation) for the type of Primer and cycling used.(NC = no cycling; TC= thermocycling; TMC=thermo mechanical cycling)

	XT Transbond			Plus SEP Transbond		
	NC	TC	TMC	NC	TC	TMC
	11.44 \pm 5.23	11.20 \pm 4.88	11.19 \pm 3.44	12.26 \pm 2.80	14.99 \pm 4.38	16.84 \pm 5.95
		11.27 \pm 4.48			14.70 \pm 4.85	

Table 3 - Two-way ANOVA, * $p < 0.05$

Effect	Df	Sum of Squares	Mean Square	F	p^*
Adhesive	1	263.82	263.819	12.62	0.0006
Cycling	2	70.67	35.334	1.69	0.1908
Adhesive*Cycling	2	89.06	44.528	2.13	0.1253
Residue	84	1756.50	20.911		
Total	89	2180.04			

Table 4 - Statistical comparison between all Groups by means of the Multiple Comparison Tukey's Test (5%).(NC = no cycling; TC= thermocycling; TMC=thermo mechanical cycling)

Primer	Cycling	Média	Grupos Homogêneos*
SEP	TMC	16.841	A
SEP	TC	14.991	AB
SEP	NC	12.263	AB
XT	NC	11.440	B
XT	TC	11.195	B
XT	TMC	11.187	B

Table 5 - Distribution of absolute and relative frequencies of ARI for groups "Type of Primer" and "Type of Cycling". (NC = no cycling; TC= thermocycling; TMC=thermo mechanical cycling)

Primer	Cycling	ARI = 0		ARI = 1		ARI = 2		ARI = 3	
SEP	NC	2	13.33	8	53.33	5	33.33	0	0.00
XT		6	40.00	5	33.33	2	13.33	2	13.33
SEP	TC	2	13.33	9	60.00	4	26.67	0	0.00
XT		9	60.00	3	20.00	3	20.00	0	0.00
SEP	TMC	2	13.33	2	13.33	7	46.67	4	26.67
XT		7	46.67	5	33.33	3	20.00	0	0.00

DISCUSSION

The aim of the current study was to determine the effects of thermal and thermomechanical cycling on SBS of conventional and self-etching primers. Various authors have accepted the SBS test performed for bovine enamel [11-14]. Some of the advantages of using it lie on the fact that it is easily standardised and also shows similar microstructure to human enamel [15]. The choice for the use of this material for the investigation was based on the studies of Yamada et al. [16]; Yamamoto et al. [17]; Cozza et al. [18] e Davari et al. [19], who considered its similarities to human enamel when submitted to SBS test.

As regards the type of acid-etching, phosphoric acid is still the best choice for surface etching. Studies show that 30% and 40% concentrations are the optimum values to produce a good retention standard to enamel [20,21]. With the introduction of acid primers which combine both acid conditioning

and primer adhesive, it has been possible for clinicians to exclude one of the steps during the bonding process, and also to minimize the amount of enamel that can be lost in post etching time. Self-etching primers do not penetrate or dissolve enamel surface at the same deepness as conventional systems which use phosphoric acid [8]. In 2003, Buyukymaz et al. [22] found greater values for SBS (16.0 ± 4.5 MPa) when using a self-conditioning primer (Transbond-Plus SEP) and compared to a conventional adhesive system - XT Transbond (13.1 ± 3.1 MPa). Values SBS found in the present study for both SEP (14.70 ± 4.85 MPa) and XT (11.27 ± 4.48 MPa) also confirm literature findings.

On the contrary, Cehrelli et al. [23], found different results when comparing SBS of 4 self-conditioning primers (Prompt L-Pop, Clearfil SE Bond, FL Bond and One-up Bond F) with a conventional adhesive system (XT Transbond). The results found were smaller for conventional systems (Prompt L-Pop 1.72 ± 0.13 MPa; Clearfil SE Bond 1.75 ± 0.19 MPa;

FL Bond 1.71 ± 0.22 MPa; One-Up Bond 1.77 ± 0.14 MPa; XT Control 10.5 ± 0.86 MPa). The authors concluded that self-etching adhesives showed the smallest values of SBS compared to the products used in the conventional groups.

Orthodontic adhesive failure can occur due to a variety of factors, e.g., saliva contamination during bonding. However, the majority of the *in vitro* studies of SBS did not take into account the influence of change in temperature in a wet environment. Several authors performed a thermal cycling process previous to SBS, in order to simulate stress in the adhesive interface, and produce a similar real clinical condition for the studied materials [8,24,25]. In the present study 500 thermal cyclings were performed in between 5 °C to 55 °C to simulate fatigue at the

adhesive interface and intra-oral conditions. Bishara et al., 2003 [8], evaluated the effect of thermal cycling in the shearing resistance of a cyanoacrylate adhesive system, 24 h post bonding and post thermal cycling. The cyanoacrylate adhesive showed a greater SBS at 24 h (7.1 ± 3.3 MPa) compared to the group which went through 500 cycles of thermal cycling between 5 °C to 55 °C (1.5 ± 1.4 MPa). These results demonstrated that the adhesive system tested shows adequate clinical adhesive resistance after 24 h of bonding, though it loses 80% of resistance in the post thermal cycling. In contrast, the present study did not show a significant decrease in bonding strength for the groups in which thermal cycling was carried out, for both XT and SEP groups.

Turk et al. [26], obtained opposite results to those found in the present work, demonstrating that thermal cycling can significantly decrease the bond strength of a self-etching primer. However, in this study, the authors performed 10 000 to 20 000 thermal cycles. The resistance values decrease was statistically different from the values obtained in thermal cycling.

Elekdg-Turk [27], also evaluated SBS of orthodontic brackets under thermal cycling in the adhesive resistance of a self-etching primer after 0, 2000 and 5000 thermal cycles. Those authors compared SBS of a conventional system - 37% acid etching with a self-etching system. Similar to this work, thermal cycling did not reveal significant difference for the conventional adhesive system group. For the self-etching conditioning group the bond strength values decreased for both 2000 and 5000 thermal cycles, and these conditions were significantly different for the non-thermal cycled group ($p < 0.001$). These results are in contrast to the findings of this work, which did not reveal a significant increase in SBS of SEP thermal cycled (14.99 ± 4.38 MPa) compared to the SEP non-thermal cycled (12.26 ± 2.80 MPa) group.

Yamamoto et al. [17] evaluated the effect of post-bonding time (5, 10, 60 min and 24 h) when using different types of orthodontic adhesives. The mean values of bonding for all systems increased with the storage period, though the increasing amount varied between the different materials used. The authors concluded that the bonding force for all investigated adhesives increased with storage time. Similar results were reported by Di Nicoló et al. [1], which by means of the ANOVA test showed that there were significant differences between the post-bonding time, and that the adhesive resistance found in the SBS test, in a period of 7 days, showed the greatest bond strength value within the periods investigated (immediately and 24 h post-bonding).

Those findings can establish a relation with the present study, mainly for the thermal cycled group. Our hypothesis stated that this group would show smaller values of shearing resistance as it would be exposed to fatigue in the adhesive interface. However, the groups which underwent thermal cycling demonstrated greater resistance values. 100 000 thermal mechanical cycles (1 bar intensity and temperature varying between 5 °C to 55 °C) were performed. All cycles spent

about 48 h to be concluded. For that matter, the shearing test for this group was performed two days after bracket bonding, which may explain the resistance increase reported before. The aim of the thermomechanical cycling was to reproduce one year of clinical treatment, simulating masticatory conditions and the change in intra-oral temperature. Eliades and Brantley [28] by means of a literature review showed the inconsistency in protocols of orthodontic bonding and proposed a new discussion to study materials fatigue, aiming to understand the processes occurring previous to adhesive failure. The main idea was to show that occlusal force simulations (a combination of compressive, shear and tension forces) on orthodontic materials used for in vitro studies could describe a real clinical performance. Thus, the choice for the thermomechanical cycling was to induce fatigue in the adhesive interface, so that the stress would simulate intra-oral conditions that could be able to reduce the enamel adhesion of tested materials. Nevertheless, the results of the present study indicate that 100 000 cycles (1 bar) were not sufficient to decrease and degrade bonding strength. Additionally, it was also observed that under stress conditions the SEP/TMC group revealed optimum values of bonding resistance (16.84 ± 5.95 MPa) and may be perfectly indicated for bracket bonding.

With regards to the ARI, it was observed that the best bonding interface pattern was the one found for SEP/ TMC, which demonstrates that even under fatigue, the self-etching primer shows a good bonding strength. The majority of the specimens showed ARI = 1 and revealed that at least a small quantity of adhesive remained on the enamel's surface. In a few samples there were no adhesive in the post-shearing, which reinforces the findings of Di Nicoló et al. [1]. The use of self-etching adhesive might favour this condition, which demonstrates smaller risk of enamel's surface loss during bracket removal.

CONCLUSION

- The number of cycles in thermal and thermo mechanical cycling did not influence shear bond strength values for any of the primers tested;
- SEP Transbond Plus showed the greatest values for bond strength for orthodontic brackets bonding to bovine enamel.

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