

Influence of Thermal and Mechanical Load Cycling on Microtensile Bond Strengths of Total and Self-etching Adhesive Systems

FHO Mitsui • AR Peris • AN Cavalcanti
GM Marchi • LAF Pimenta

Clinical Relevance

Bond strength is affected by the combination of thermal and mechanical load cycling. However, results vary greatly with the number of mechanical cycles and adhesive system type (total or self-etching).

SUMMARY

This study evaluated the influence of different thermal (TC) and mechanical (MC) cycling protocols on microtensile bond strength (μ TBS) to cervical dentin margins of Class II restorations using two total-etch (TE) adhesives and one self-

etching (SE) primer. Class II slot cavities were prepared on the mesial surfaces of 168 bovine incisors and were divided into three groups according to the bonding system used: Single Bond, OptiBond Solo Plus and Clearfil SE Bond. All cavities were restored with Filtek Z250 composite. Following restorative procedures, the restored teeth were allocated to seven subgroups (n=8) according to the thermal/mechanical protocol performed: G1-control (no cycling), G2-100,000 MC, G3-200,000 MC, G4-500,000 MC, G5-100,000 MC+1,000 TC, G6-200,000 MC+1,000 TC, G7-500,000 MC+1,000 TC. TC was performed using $5 \pm 2^\circ\text{C}$ and $55 \pm 2^\circ\text{C}$ baths, with a dwell time of 60 seconds in each bath. MC was achieved with an axial force of 80 N at 2 cycles/second. The restorations were sectioned perpendicular to the cervical bonded interface into two 0.8-1-mm thick slabs. The slabs were trimmed at the interface to obtain a cross-sectional surface area of 0.8-1 mm². All specimens were then subjected to μ TBS ($v=0.5$ mm/minute). Fracture mode analysis was performed using SEM. Bond strength mean values (MPa) were analyzed with ANOVA 3-way and Tukey's test ($\alpha=5\%$). Dunnett's test was used to compare tested groups against Control groups of

*Fabio Hiroyuki Ogata Mitsui, DDS, MS, PhD, assistant professor, Department of Restorative Dentistry, University of State of Amazon (UEA), Manaus, AM, Brazil

Alessandra Rezende Peris, DDS, MS, PhD, assistant professor, Department of Restorative Dentistry, University of State of Amazon (UEA), Manaus, AM, Brazil

Andrea Nóbrega Cavalcanti, DDS, MS, graduate student of PhD program, Department of Restorative Dentistry, University of Campinas, School of Dentistry of Piracicaba, Sao Paulo, Brazil

*Giselle Maria Marchi, DDS, MS, PhD, assistant professor, Department of Restorative Dentistry, University of Campinas, School of Dentistry of Piracicaba, Sao Paulo, Brazil

Luiz André Freire Pimenta, DDS, MS, PhD, full professor of Restorative Dentistry, Department of Restorative Dentistry, University of Campinas, School of Dentistry of Piracicaba, Sao Paulo, Brazil

*Reprint request: Av Carvalho Leal, 1777, Cachoeirinha, Manaus, AM, Brazil: e-mail: fabio_mitsui@yahoo.com.br

DOI: 10.2341/05-20

each adhesive system ($\alpha=5\%$). SE primer presented lower mean bond strength values when compared to TE adhesives ($p=0.05$). In addition, specimens restored with the SE primer did not resist to the 200,000 and 500,000 MC associated with TC. The application of 100,000 MC did not present a significant decrease in bond strength when compared to the control. Mixed failures were predominant for all groups. The higher the amount of thermal/mechanical cycles, the greater the number of mixed failures and the lower the percentage of adhesive failures.

INTRODUCTION

The increasing demand for esthetic restorations in daily clinical dentistry, as an alternative to amalgam restorations, has generated intensive research in adhesive materials (Perdigão & others, 1996). An adequate tooth/restoration interface in composite restorations would produce well-sealed, long-lasting restorations and would allow for a more conservative approach to treating dental lesions (Perdigão & others, 1996). To help maintain the integrity of the restoration, the adhesive interface must resist dimensional changes to prevent developing leakage and possible further degradation of the restoration (Leibrock & others, 1999), which could also occur as a result of chemical, thermal and mechanical load stresses (Abdalla & Davidson, 1993; da Cunha Mello & others, 1997).

Dentin adhesives have been widely used in clinical dentistry, and different versions and commercial brands are constantly being introduced, claiming advantages over their predecessors (Tanumiharja, Burrow & Tyas, 2000). A clinical trial is the most effective method to assess the quality of these materials (Tanumiharja & others, 2000). However, due to the time it takes for long-term clinical data to become available, products are often superseded (Tanumiharja & others, 2000). Therefore, the application of *in vitro* methodologies using different types of stresses to simulate the aging of restorations has been advocated. These stressing tests could accelerate deterioration of the dentin/restoration interface, (DeLong & Douglas, 1983; Abdalla & Davidson, 1993; da Cunha Mello & others, 1997) and enable better evaluation of the behavior of adhesive materials exposed to stresses similar to those found in the oral environment.

The use of thermal cycling (TC) in dental restorations is frequently seen in laboratory studies in order to simulate changing intraoral temperature conditions (Bedran-de-Castro & others, 2004a,b). According to some authors (Rossomando & Wendt Jr, 1995; Gale & Darvell, 1999), the difference in the coefficient of thermal expansion between tooth structure and restorative materials might induce degradation of the dentin/restoration interface.

Mechanical cycling (MC) has been studied due to its potential capability of simulating mastication (Bedran-de-Castro & others, 2004a,b). According to da Cunha Mello and others (1997), the application of occlusal MC could increase the vulnerability of the restoration-cavity wall interface, which is already stressed by the curing contraction. However, as with TC, it is difficult to compare studies since they employ different loads, number of cycles and cycle frequency (Bedran-de-Castro & others, 2004a).

This study investigated the effect of different MC protocols in association with TC on microtensile bond strength (μ TBS) and also evaluated failure patterns at the cervical dentin margins of composite Class II slot preparations restored with two total-etch (TE) adhesive systems and one self-etching (SE) primer. The null hypotheses tested were that there was no relationship between mechanical and thermal cycling to microtensile shear bond strength.

METHODS AND MATERIALS

One hundred and sixty eight extracted bovine incisors were collected and stored in a 0.1% sodium azide saline solution. The teeth were scaled with periodontal curettes (Duflex, Rio de Janeiro, RJ, Brazil) and cleaned with water. Incisal surfaces were horizontally sectioned 5.0 mm above the CEJ (Figures 1A and 1B) using a double-faced diamond disc (KG Sorensen, Barueri, SP, Brazil), allowing for configuration of a flat standard "occlusal" surface.

Class II slot preparations were prepared on the mesial surface (Figure 1C) with a high-speed #245 carbide bur (KG Sorensen) under constant water cooling. The cavity preparations were 4.0-mm wide, 6.0-mm high (1.0 mm below the CEJ) and 1.5-mm deep toward the pulp chamber. In addition, the angles formed between all cavity walls were rounded (Figure 1C). The burs were replaced after every five preparations.

Cavities were randomly allocated to three distinct groups according to the adhesive system used (Table 1). The application of each adhesive system followed the manufacturers' instructions (Table 1). All cavities were restored with Filtek Z250 composite (3M ESPE Dental Products, St Paul, MN, USA) in three 2.0 mm horizontal increments and light-cured for 20 seconds each. A 1.0 mm overfill was left on the occlusal surface for MC testing. This 1.0 mm overfill was then finished and polished with Al_2O_3 abrasive discs (Sof-Lex Pop-on/3M ESPE Dental Products) to a flat, smooth surface. During all restorative procedures, the QTH intensity (VIP Jr, BISCO Dental Products, Schaumburg, IL, USA) was measured by a radiometer (Hilux Light Meter; First Medica, Greensboro, NC, USA) and ranged from 700 to 760 mW/cm². After the restorative procedure, the specimens were stored in distilled water at 37°C for 24 hours.

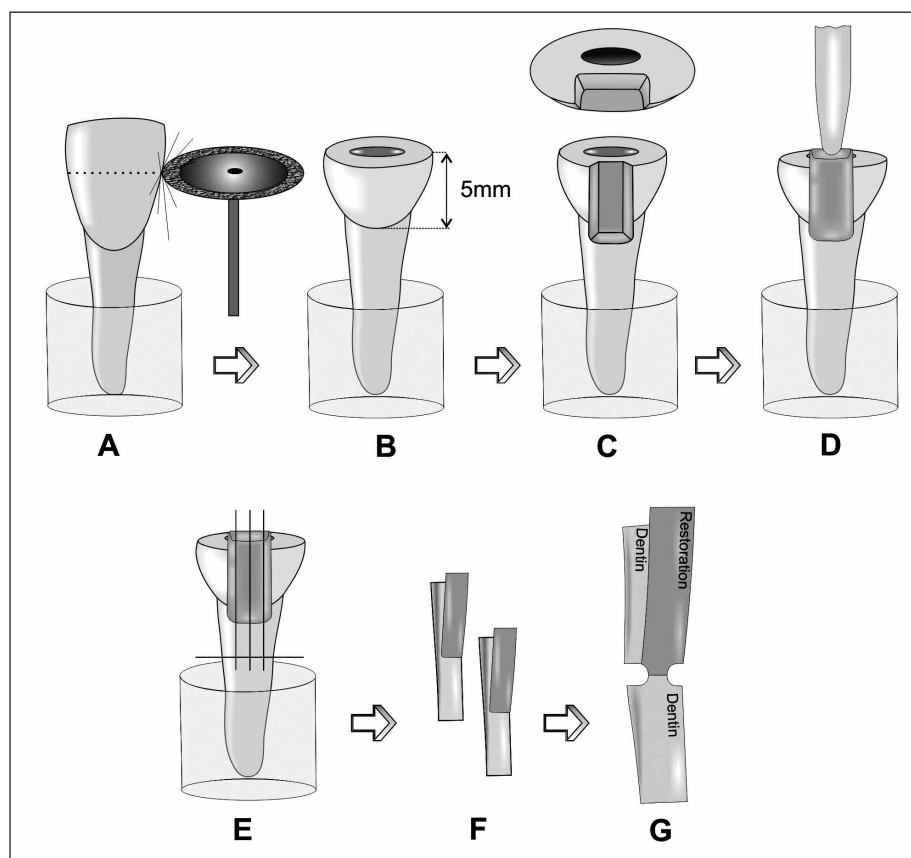


Figure 1. Tooth sectioning with a double-faced diamond disc (A), 5.0 mm above the CEJ (B); Class II slot preparations concluded, 1.0 mm below the CEJ—proximal and occlusal-gingival directions (C); Load application on the flat overfilled “occlusal” surface (D); Restoration sectioning (E) and slab configuration for microtensile bond strength test (F,G).

The restored specimens were then allocated into seven subgroups (n=8):

- G1—Control (no TC and MC)
- G2—MC (100,000x)
- G3—MC (200,000x)
- G4—MC (500,000x)
- G5—TC (1,000x) and MC (100,000x)
- G6—TC (1,000x) and MC (200,000x)
- G7—TC (1,000x) and MC (500,000x)

The specimens subjected to MC had part of their roots embedded in cold cure polystyrene resin (Cromex, Piracicaba, SP, Brazil) in order to keep the flat standard occlusal surface perpendicular to the long axis of the tooth (Figure 1D).

Thermal and Mechanical Load Cycling

The MC test was conducted in a Mechanical Loading Machine (ERIOS Representações e Comércio Ltda, Sao Paulo, SP, Brazil) with a 15.0-mm cylindrical metallic tip attached to a steel bar placed in contact with the restoration (Figure 1D). The loading device delivered an

intermittent axial force of 80 N at 2 cycles/second.

Specimens from groups G5 to G7 were also subjected to additional 1,000 TC in thermocycling apparatus (MCT2-AMM-2 Instrumental, Sao Carlos, SP, Brazil) with two baths at $5 \pm 2^\circ\text{C}$ and $55 \pm 2^\circ\text{C}$ each and a dwell time of 60 seconds.

Microtensile Bonding Test

The restorations were sectioned perpendicular to the cervical bonded interface of each tooth (Figure 1E) into serial 0.7 ± 0.2 -mm thick slabs (n=2 per restoration) with a slow speed diamond wafering blade (Buehler, Lake Bluff, IL, USA) and constant water coolant. These slabs (Figure 1F) were then trimmed and shaped to form a gentle curve with the narrowest portion at the composite/dentin interface (Figure 1G) and standardized to produce a bonded surface area of $1.0 \pm 0.2 \text{ mm}^2$ using a #1093FF fine diamond bur (Injecta, Diadema, SP, Brazil). All specimens were then mounted in testing apparatus with a cyanocrylate adhesive (Super Bonder, Henckel Loctite, Itapevi, SP, Brazil) and debonded using a universal testing machine (DL 500; EMIC Ltda, Sao Jose dos Pinhais, PR, Brazil) at a crosshead speed of 0.5 mm/minute until failure. Means and standard deviations were calculated and expressed in MPa. Statistical analysis was performed using 3-way ANOVA and Tukey's test at a significance level of 5%. Dunnett's test was used to compare the groups that were submitted to MC and TC, with the Control group of each adhesive system ($\alpha=5\%$).

Fracture Mode Analysis

Following μTBS test, all fractured specimens were stored in 10% neutral buffered formalin solution for at least eight hours after debonding. The fractured surfaces were then dried, mounted on aluminum stubs, gold sputter-coated (Denton Vacuum Desk II; Denton Vacuum LLC, Moorestown, NJ, USA) and observed with a scanning electron microscope (JSM 5600 LV; JEOL, Tokyo, Japan) to evaluate the fracture pattern. The predominant fracture modes were classified into one of four types (Bedran-de-Castro & others, 2004b): adhesive failure (Type 1) observed at the interphase located between the deepest portion of the adhesive resin and the top layer of the demineralized dentin (Nakabayashi & Pashley, 1998), cohesive failure in

| Table 1: Description of the Adhesive Systems Used in this Study, Their Batch Number, Composition, and Manufacturers' Instructions of the Sequence of Adhesive Systems Application | | |
|---|--|---|
| Adhesive System (Batch #) | Components | Procedures |
| Single Bond 3M ESPE St Paul, MN, USA (3HY) | Ethanol, Bis-GMA, HEMA, water, polyalkenoic acid, acid copolymer. | The dentin surface was etched with H ₃ PO ₄ 35%, rinsed with water for 15 seconds and gently air dried for 2 seconds; two consecutive coats of the adhesive were applied, lightly air dried for 2 seconds, and light-cured for 10 seconds. |
| Optibond Solo Plus Kerr Corp, Orange, CA, USA (304142) | Ethanol, Bis-GMA, HEMA, GPDM, silica, barium glass, sodium hexafluorosilicate. | The adhesive bottle was shaken before using. The dentin surface was etched with 35% H ₃ PO ₄ , rinsed with water for 15 seconds and gently air dried for 2 seconds; one coat of the adhesive was applied, rubbed for 15 seconds and light-cured for 20 seconds. |
| Clearfil SE Bond Kuraray, Osaka, Japan (Primer-00410A) (Bond-00547A) | <i>Primer:</i> N,N-Diethanol-p-toluidine, MDP, HEMA, hydrophilic dimetacrylate, DL-camphorquinone, water. <i>Bond:</i> N,N-Diethanol-p-toluidine, MDP, Bis-GMA, HEMA, hydrophobic dimetacrylate, DL-camphorquinone, silanated colloidal silica. | Primer was applied for 20 seconds and gently air dried for 2 seconds; one coat of the adhesive was applied, gently air dried for 2 seconds and light-cured for 10 seconds. |

| Table 2: Means (MPa) and Standard Deviation for Microtensile Bond Strength Values Evaluated by the Tukey's Test ($p < 0.05$) | | | | |
|--|-----------------|------------------------------------|------------------------------------|------------------------------------|
| Mechanical Cycling | Thermal Cycling | Single Bond | Optibond Solo Plus | Clearfil SE Bond |
| 100,000 | - | 32.61 \pm 6.83 a | 27.63 \pm 4.63 a | 24.21 \pm 6.78 a |
| 100,000 | + | 25.86 \pm 7.39 b | 25.87 \pm 5.36 b | 20.08 \pm 5.39 b |
| 200,000 | - | 26.09 \pm 4.32 a | 26.29 \pm 9.28 a | 19.95 \pm 1.78 |
| 200,000 | + | 26.48 \pm 4.30 a | 27.27 \pm 5.37 a | --- |
| 500,000 | - | 26.42 \pm 3.36 a | 26.13 \pm 4.98 a | 21.09 \pm 4.87 |
| 500,000 | + | 31.94 \pm 4.58 a | 25.74 \pm 3.82 a | --- |
| CONTROL | CONTROL | 40.41 \pm 8.48 | 34.34 \pm 6.68 | 26.65 \pm 8.41 |
| | | A | A | B |
| (+ = performed / - = not performed) (--- = specimens lost during slab trimming procedure) Statistically significant differences are expressed by capital letters in columns and by lower case letters in rows ($p < 0.05$) | | | | |

composite (Type 2), cohesive failure in adhesive (Type 3) and mixed failure (association of two or more failures) (Type 4). The percentage of each failure mode was calculated from the frequency observed in each experimental group.

RESULTS

Microtensile Bond Strength

Tukey's test (Table 2) did not show statistically significant differences between the two TE adhesive systems tested. However, the SE primer Clearfil SE Bond presented significantly lower bond strength values ($p = 0.05$) when compared to both TE adhesives (Single Bond and Optibond Solo Plus). In addition, it is important to state that all specimens restored with SE primer and submitted to 200,000 MC + TC (G6) and 500,000 MC + TC (G7) were lost during slab trimming procedures.

Statistically significant interactions between adhesive systems and TC ($p = 0.053$) was observed. For all

adhesive systems, bond strengths were significantly lower when TC was performed on the specimens submitted to 100,000 MC (Table 2). However, TC had no significant influence on the specimens submitted to 200,000 (G6) and 500,000 MC (G7).

Dunnett's test (Table 3) allowed for comparison between the groups that were submitted to TC and MC, with the Control group (untreated) of each adhesive system. For the TE Single Bond adhesive system, the application of 100,000 MC did not show significant differences. All other cycling protocols presented lower μ TBS when compared to the Control group. For the TE Optibond Solo Plus adhesive system, a statistically significant difference was only observed for the specimens subjected to 500,000 MC + TC. Finally, for SE primer Clearfil SE Bond, all groups submitted to TC or MC did not present any significant difference from the Control group.

Fracture Mode Analysis

The fractured pattern was evaluated in all specimens and the results are presented in Table 4. The four types

| Mechanical Cycling | Thermal Cycling | Single Bond | Optibond Solo Plus | Clearfil SE Bond |
|--------------------|-----------------|--------------|--------------------|------------------|
| 100,000 | - | 32.61 | 27.63 | 24.21 |
| 100,000 | + | 25.86* | 25.87 | 20.08 |
| 200,000 | - | 26.09* | 26.29 | 19.95 |
| 200,000 | + | 26.48* | 27.27 | --- |
| 500,000 | - | 26.42* | 26.13 | 21.09 |
| 500,000 | + | 31.94* | 25.74* | --- |
| CONTROL | | 40.41 | 34.34 | 26.65 |

(+ = performed / - = not performed)
 (--- = specimens lost during slab trimming procedure)
 * indicates statistically significant difference in comparison with Control groups

| | | G1 | G2 | G3 | G4 | G5 | G6 | G7 |
|---------------------------|--------|-------|----|-------|-------|-------|-------|-------|
| Single Bond | Type 1 | 31.25 | 25 | 18.75 | 18.75 | 18.75 | 6.25 | 6.25 |
| | Type 2 | 0 | 0 | 0 | 0 | 6.25 | 6.25 | 12.5 |
| | Type 3 | 6.25 | 0 | 6.25 | 0 | 0 | 0 | 0 |
| | Type 4 | 62.5 | 75 | 75 | 81.25 | 75 | 87.5 | 81.25 |
| Optibond Solo Plus | Type 1 | 18.75 | 25 | 25 | 18.75 | 6.25 | 6.25 | 6.25 |
| | Type 2 | 0 | 0 | 0 | 0 | 12.5 | 12.5 | 12.5 |
| | Type 3 | 18.75 | 0 | 0 | 0 | 12.5 | 0 | 12.5 |
| | Type 4 | 62.5 | 75 | 75 | 81.25 | 68.75 | 81.25 | 68.75 |
| Clearfil SE Bond | Type 1 | 50 | 25 | 25 | 18.75 | 18.75 | --- | --- |
| | Type 2 | 0 | 0 | 0 | 6.25 | 6.25 | --- | --- |
| | Type 3 | 6.25 | 0 | 0 | 0 | 0 | --- | --- |
| | Type 4 | 43.75 | 75 | 75 | 75 | 75 | --- | --- |

G1-control (no cycling), G2-100,000 MC, G3-200,000 MC, G4-500,000 MC G5-100,000 MC+1,000 TC, G6-200,000 MC+1,000 TC, G7-500,000 MC+1,000 TC.
 Type 1—adhesive failure/Type 2—cohesive failure in composite/Type 3—cohesive failure in adhesive/Type 4—mixed failure
 (--- = specimens lost during slab trimming procedure)

of failure modes (adhesive, cohesive in composite, cohesive in adhesive and mixed) were observed. Mixed failures (Type 4) were predominant for all groups. Adhesive failure (Type 1) was also verified in all groups, but especially in the Control groups. The number of Type 4 failures increased, and the percentage of Type 1 failures decreased when the amount of thermal/mechanical cycles applied was higher. Cohesive failure in resin (Type 2) was only observed when TC was performed (G5-G7). Cohesive failures in adhesive (Type 3) were observed in a very low percentage, especially in teeth restored with Optibond Solo Plus.

DISCUSSION

The rapid development of adhesive restorative materials has allowed for significant improvement in restorative procedures. Unfortunately, the continuous and fast progress of adhesive restorative materials, combined with high costs and the immediate demand for information, does not allow for long-term clinical trials (Tanumiharja & others, 2000). Consequently, the establishment of *in vitro* methodologies simulating oral conditions constitutes an important means of evaluating

the clinical potential of these materials (da Cunha Mello & others, 1997; Nikaido & others, 2002a).

MC is based on the application of repeated load cycles, thus simulating a clinical masticatory process (Bedran-de-Castro & others, 2004a,b). In this study, the use of a cylindrical metallic tip touching only the restorative material was intended to fatigue the restoration. Some studies have reported the effects of MC on μ TBS (Nikaido & others, 2002a,b; Bedran-de-Castro & others, 2004a,b). However, these studies present a great variation in methodology with regard to applied force (50 N [Bedran-de-Castro & others, 2004b]—125 N [Abdalla & Davidson, 1993]) and, primarily, the number of cycles (4000 [Abdalla & Davidson, 1993; da Cunha Mello & others, 1997]—100,000 [Nikaido & others, 2002a]) used. The load of 80 N was chosen as an average of the masticatory forces observed by Anderson (1956) and was also applied in recent studies (Mitsui & others, 2003; Bedran-de-Castro & others, 2004a).

The laboratory simulation of 100,000 MC on Class II slot restorations performed in this study did not present a statistically significant difference in bond strength

when compared with the Control groups (Table 3), despite of the adhesive system evaluated. These results are in accordance with the results of Nikaido and others (2002b) and Bedran-de-Castro and others (2004b), who also found a lack of influence of 100,000 MC on μ TBS. It could be speculated that when this MC count was performed alone, the dentin/restoration interface formed by the adhesive systems evaluated in this study did not deteriorate.

The application of TC as a method for aging specimens in *in vitro* tests has been thoroughly investigated (Burger, Cooley & Garcia-Godoy, 1992; Nikaido & others, 2002a; Bedran-de-Castro & others, 2004a,b). The number of cycles and the temperatures used seem to be major differences among the studies. According to results from the Burger and others (1992) study, the application of a great number of TCs did not lead to a decrease in bond strength. Thus, 1,000 cycles were used as an average from recent articles, and the use of ISO standardized bath temperatures allowed for a comparison among studies.

It is important to state that, in the group where TC was performed concurrently with 100,000 MC (G5), mean bond strength values were significantly lower (Table 2). According to Nikaido and others (2002a), deformation of the restoration can take place after MC and TC, which may cause micro-separations to develop between the cavity floor and the adhesive or plastic deformation of the adhesive interface. However, TC had no significant influence on the specimens also submitted to 200,000 (G6) and 500,000 MC (G7). The authors suggest that the magnitude of the action performed by those amounts of MC (200,000 and 500,000) had already been enough to deteriorate the adhesive interface, thus lessening the TC effect. According to the fracture mode analysis, the use of TC was responsible for the cohesive failures in resin (Type 2) observed in this study, suggesting that the high temperature gradient applied in this laboratory test might have altered the mechanical properties of the restorative composite used in this study.

For the TE adhesive system (Single Bond), the statistically significant influence of thermal/mechanical treatment on μ TBS could be observed, except for the group submitted to only 100,000 MC (Table 3). A possible explanation for the lower results associated with higher mechanical/thermal cycling magnitude might be the adhesive composition. Bis-GMA and HEMA are the main chemical components of Single Bond (Table 1). According to Xu and others (1997), HEMA solution reacts with the collagen fibrils in dentin due to hydrogen bonds or new bonds to ester groups. However, it has been speculated that these chemical bonds are fragile. Thus, the action of the hydrogen bond might be reduced by thermal/mechanical tensions or by the presence of

intrinsic water contained within the collagen fibrils in the moist bonding technique (Hashimoto & others, 2001). The use of a moist bonding technique in cavity preparations makes blot drying a critical step, and the presence of excess water along line angles could compromise full evaporation of the solvent (Zheng & others, 2001), resulting in a reduction in the degree of monomer conversion, hence lowered bond strength (Jacobsen & Söderholm, 1995; Pereira & others, 1999).

Furthermore, it is worth remembering that the success of bonding depends mainly on penetration capability of the adhesive resin into the exposed collagen network in order to create the hybrid layer (Nakabayashi, Kojima & Masuhara, 1982). However, according to Phrukkanon, Burrow and Tyas (1999), in general, the adhesive was unable to fully penetrate into the base of the exposed collagen, creating porous zones along the base of the hybrid layer. These porous zones might be susceptible to hydrolysis after TC, therefore explaining the decrease of Single Bond μ TBS (Miyazaki & others, 1998). These same porous zones were also observed in this study during the fracture mode analysis.

For the Optibond Solo Plus TE adhesive system, statistically significant differences were only observed for the specimens submitted to the protocol of 500,000 MC +TC (Table 3). A descriptive hypothesis of this result could be that TC, applied prior to MC, might have interfered with μ TBS. The subsequent application of loading with great magnitude (500,000 cycles) to an interface that had previously undergone thermal exposure might have increased the effects of loading on the tooth/restoration interface, thus explaining the decrease in μ TBS.

Polymerization shrinkage of composites is the most relevant concern faced in achieving a successful restorative procedure, since the stress generated by this contraction challenges the bonded-interface area (Montes & others, 2001). Filler has been added to adhesives in order to improve bond strengths by reinforcing the hybrid layer and reducing polymerization shrinkage (Gallo & others, 2001). The TE adhesive system Optibond Solo Plus contains approximately 26% barium glass filler (0.6 μ m) in its composition (Table 1). This amount of filler gives the bond a gel consistency, thus forming a low elastic modulus hybrid layer that could work as a stress absorbing layer and also allow for a more homogeneous distribution of thermal and mechanical stresses (Braga, Cesar & Gonzaga, 2000; Gallo & others, 2001; Montes & others, 2001). The association of these factors might explain the lack of statistically significant difference among the groups submitted to 100,000 or 200,000 MC, with or without TC, compared with the Control group.

The greatest frequency of cohesive failures in adhesive (Type 3) was observed in the specimens restored

with TE Optibond Solo Plus adhesive. A possible explanation for this finding might be its viscosity. Due to its higher filler content, this system allows for the formation of a thicker adhesive layer that could work as a weak point when submitted to load forces (Hashimoto & others, 2001). Furthermore, the brush motion utilized during application of Optibond Solo Plus also could include air bubbles within the adhesive layer (Montes & others, 2001), which might also work as a weak point for fracturing after loading.

SE primers have been designed to simplify bonding procedures by eliminating separate acid etching and avoid the risk of overetching, overdrying or overwetting the dentin (Cardoso & others, 2001; Molla, Park & Haller, 2002). The mechanism of the SE primers is characterized by the simultaneous demineralization and resin infiltration of the substrate (Watanabe, Nakabayashi & Pashley, 1994; Molla & others, 2002). Some *in vitro* studies reported similar dentin bond strengths of SE primers and TE adhesive systems (Cardoso & others, 2001). However, in this study, the SE primer presented significantly lower bond strength ($p=0.05$) than that observed by both TE adhesives evaluated in this study (Single Bond and Optibond Solo Plus). These results are in accordance with the results from Hashimoto and others (2001) and Chaves, Giannini and Ambrosano (2002), who also reported lower bond strength values for SE primers.

For those specimens that were restored with SE primer and submitted to the protocol of only 100,000 MC or the association of 100,000 MC + TC, no statistically significant difference was observed (Table 3). The results of this study are in accordance with previous data from Kaaden and others (2002) and Mitsui and others (2003) studies, which did not observe the influence of MC on teeth restored using SE primer. Clearfil SE Bond contains approximately 10% particle filler in its composition. According to Kaaden and others (2002), the hybrid layer formed by this SE primer has elastic characteristics that could work to absorb stresses and also propitiate a more homogeneous distribution of thermal and mechanical stresses. Nevertheless, it is important to point out that all specimens restored with SE primer and submitted to a greater cycling stress (200,000 MC + TC and 500,000 MC + TC) were lost during slab trimming. It could be speculated that the narrow hybrid layer formed by this adhesive, approximately 0.4 to 0.5 μm (Tay & Pashley, 2001), was not resistant to the amount of stress developed by the associated thermal and mechanical cycling. In 1998, Prati and others reported no correlation between hybrid layer thickness and higher bond strength. However, it is important to emphasize that there are no reports in the literature predicting the influence of load application in narrow adhesive interfaces developed by SE primers, as verified in this study.

Fracture mode analysis showed that, independent of the adhesive system used, the higher the amount of thermal/mechanical cycles, the greater the number of mixed failures (Type 4) and the lower the percentage of adhesive failures (Type 1). Type 4 fracture mode consisted of three kinds of fractures, whether or not they appeared together. It is difficult to specify the localized stress point at the interface. Observing that the fracture at interphase was reduced with higher stress magnitude, another interesting aspect of this study is that the authors' results suggest that MC could weaken adhesive resin at the bonded interface.

Degradation of the adhesive interface due to mechanical loading is a very important issue in restorative dentistry, since patients' demand for posterior composite restorations has significantly increased. Further studies should be conducted with human teeth, different load forces and also new adhesive systems, such as self-etching adhesives, in order to improve the quality of adhesive restorative materials.

CONCLUSIONS

Within the limitations of this study, the following conclusions may be drawn:

1. The effectiveness of the cycling protocols performed in this study showed different behaviors according to the adhesive system evaluated;
2. The application of 100,000 MC by itself did not present significantly lower μTBS for all adhesive systems evaluated;
3. SE primer presented lower mean bond strength values when compared to TE adhesives. In addition, specimens restored with the SE primer did not resist to 200,000 and 500,000 MC associated with TC;
4. The higher the amount of thermal/mechanical cycles, the greater the number of mixed failures (Type 4) and the lower the percentage of adhesive failures (Type 1), suggesting that MC could weaken adhesive resin at the bonded interface.

(Received 51 February 2005)

References

- Abdalla AI & Davidson CL (1993) Comparison of the marginal integrity of *in vivo* and *in vitro* Class II composite restorations *Journal of Dentistry* **21**(3) 158-162.
- Anderson DJ (1956) Measurement of stress in mastication I *Journal of Dental Research* **35**(5) 664-670.
- Bedran-de-Castro AK, Cardoso PE, Ambrosano GM & Pimenta LA (2004a) Thermal and mechanical load cycling on microleakage and shear bond strength to dentin *Operative Dentistry* **29**(1) 42-48.

- Bedran-de-Castro AK, Pereira PN, Pimenta LA & Thompson JY (2004b) Effect of thermal and mechanical load cycling on microtensile bond strength of a total-etch adhesive system *Operative Dentistry* **29**(2) 150-156.
- Braga RR, Cesar PF & Gonzaga CC (2000) Tensile bond strength of filled and unfilled adhesives to dentin *American Journal of Dentistry* **13**(2) 73-76.
- Burger KM, Cooley RL & García-Godoy F (1992) Effect of thermal cycling times on dentin bond strength *Journal of Esthetic Dentistry* **4**(6) 197-198.
- Cardoso PE, Carrilho MR, Francci CE & Perdigão J (2001) Microtensile bond strengths of one-bottle dentin adhesives *American Journal of Dentistry* **14**(1) 22-24.
- Chaves P, Giannini M & Ambrosano GM (2002) Influence of smear layer pretreatments on bond strength to dentin *Journal of Adhesive Dentistry* **4**(3) 191-196.
- da Cunha Mello FS, Feilzer AJ, de Gee AJ & Davidson CL (1997) Sealing ability of eight resin bonding systems in a Class II restoration after mechanical fatiguing *Dental Materials* **13**(6) 372-376.
- DeLong R & Douglas WH (1983) Development of an artificial oral environment for the testing of dental restoratives: Bi-axial force and movement control *Journal of Dental Research* **62**(1) 32-36.
- Gale MS & Darvell BW (1999) Thermal cycling procedures for laboratory testing of dental restorations *Journal of Dentistry* **27**(2) 89-99.
- Gallo JR, Comeaux R, Haines B, Xu X & Burgess JO (2001) Shear bond strength of four filled dentin bonding systems *Operative Dentistry* **26**(1) 44-47.
- Hashimoto M, Ohno H, Kaga M, Endo K, Sano H & Oguchi H (2001) Fractographical analysis of resin-dentin bonds *American Journal of Dentistry* **14**(6) 355-360.
- Jacobsen T & Söderholm KJ (1995) Some effects of water on dentin bonding *Dental Materials* **11**(2) 132-136.
- Kaaden C, Powers JM, Friedl KH & Schmalz G (2002) Bond strength of self-etching adhesives to dental hard tissues *Clinical Oral Investigations* **6**(3) 155-160.
- Leibrock A, Degenhart M, Behr M, Rosentritt M & Handel G (1999) *In vitro* study of the effect of thermo- and load-cycling on the bond strength of porcelain repair systems *Journal of Oral Rehabilitation* **26**(2) 130-137.
- Mitsui FH, Bedran-de-Castro AK, Ritter AV, Cardoso PE & Pimenta LA (2003) Influence of load cycling on marginal microleakage with two self-etching and two one-bottle dentin adhesive systems in dentin *Journal of Adhesive Dentistry* **5**(3) 209-216.
- Miyazaki M, Sato M, Onose H & Moore BK (1998) Influence of thermal cycling on dentin bond strength of two-step bonding systems *American Journal of Dentistry* **11**(3) 118-122.
- Molla K, Park HJ & Haller B (2002) Bond strength of adhesive/composite combinations to dentin involving total- and self-etch adhesives *Journal of Adhesive Dentistry* **4**(3) 171-180.
- Montes MA, de Goes MF, da Cunha MR & Soares AB (2001) A morphological and tensile bond strength evaluation of an unfilled adhesive with low-viscosity composites and a filled adhesive in one and two coats *Journal of Dentistry* **29**(6) 435-441.
- Nakabayashi N, Kojima K & Masuhara E (1982) The promotion of adhesion by the infiltration of monomers into tooth substrates *Journal of Biomedical Material Research* **16**(3) 265-273.
- Nakabayashi N & Pashley DH (1998) Evolution of dentin-resin bonding in: *Hybridization of Dental Hard Tissues* Quintessence Publishing Co Ltd Tokyo 1-17.
- Nikaido T, Kunzelmann KH, Chen H, Ogata M, Harada N, Yamaguchi S, Cox CF, Hickel R & Tagami J (2002a) Evaluation of thermal cycling and mechanical loading on bond strength of a self-etching primer system to dentin *Dental Materials* **18**(3) 269-275.
- Nikaido T, Kunzelmann KH, Ogata M, Harada N, Yamaguchi S, Cox CF, Hickel R & Tagami J (2002b) The *in vitro* dentin bond strengths of two adhesives systems in Class I cavities of human molars *Journal of Adhesive Dentistry* **4**(1) 31-39.
- Perdigão J, Lambrechts P, Van Meerbeek B, Braem M, Yildiz E, Yucel T & Vanherle G (1996) The interaction of adhesive systems with human dentin *American Journal of Dentistry* **9**(4) 167-173.
- Pereira PN, Okuda M, Sano H, Yoshikawa T, Burrow MF & Tagami J (1999) Effect of intrinsic wetness and regional difference on dentin bond strength *Dental Materials* **15**(1) 46-53.
- Phrukkanon S, Burrow MF & Tyas MJ (1999) The effect of dentine location and tubule orientation on the bond strengths between resin and dentine *Journal of Dentistry* **27**(4) 265-274.
- Prati C, Chersoni S, Mongiorgi R & Pashley DH (1998) Resin-infiltrated dentin layer formation of new bonding systems *Operative Dentistry* **23**(4) 185-194.
- Rossomando KJ & Wendt SL Jr (1995) Thermocycling and dwell times in microleakage evaluation for bonded restorations *Dental Materials* **11**(1) 47-51.
- Tanumiharja M, Burrow MF & Tyas MJ (2000) Microtensile bond strengths of seven dentin adhesive systems *Dental Materials* **16**(3) 180-187.
- Tay FR & Pashley DH (2001) Aggressiveness of contemporary self-etching systems. I: Depth of penetration beyond dentin smear layers *Dental Materials* **17**(4) 296-308.
- Watanabe I, Nakabayashi N & Pashley DH (1994) Bonding to ground dentin by a Phenyl-P self-etching primer *Journal of Dental Research* **73**(6) 1212-1220.
- Xu J, Stangel I, Butler IS & Gilson DF (1997) An FT-Raman spectroscopic investigation of dentin and collagen surfaces modified by 2-hydroxyethylmethacrylate *Journal of Dental Research* **76**(1) 596-601.
- Zheng L, Pereira PN, Nakajima M, Sano H & Tagami J (2001) Relationship between adhesive thickness and microtensile bond strength *Operative Dentistry* **26**(1) 97-104.