Shear Bond Strength of Different Adhesive Systems to Primary Dentin and Enamel

Carla Miranda* / Luiz Henrique Maykot Prates** / Ricardo de Souza Vieira***
Maria Cristina Marino Calvo****

The aims of this study were to evaluate the shear bond strength (SBS) of four adhesive systems applied to primary dentin and enamel and verify, after SBS testing, the failure mode of the adhesive interface. Sixty extracted sound primary molars were selected and crowns were sectioned in a mesial-distal direction. Specimens were randomly assigned into two groups (adhesion to enamel and adhesion to dentin) and then subdivided into four subgroups according to the adhesive system (n=15): Scotchbond Multi-Purpose (SMP) – Single Bond (SB) – Clearfil SE Bond (and Adper Prompt L-Pop (APL) – SBS tests were performed and the obtained values were statistically analyzed using ANOVA and Tukey tests (p<0.05). The failure mode analysis was performed with a Scanning Electron Microscope (XL-30, Philips). SBS mean values on enamel were [MPa (SD)]: SMP – 27.89 (7.49); SB – 23.92 (8.8); CSB – 24.36 (6.69); APL – 25.96 (4.08); and on dentin: SMP – 17.29 (4.25); SB – 18.2 (8.74); CSB – 16.13 (7.14); APL – 6.04 (3.35). The predominant failure mode was cohesive (primarily of the bonding agent). On enamel SBS was statistically similar for all four adhesives. On dentin SBS of APL was lower than the other tested adhesives.

Key words: *deciduous tooth, shear strength, dentin-bonding agents, dental materials, pediatric dentistry*
J Clin Pediatr Dent 31(1):35-40, 2006

INTRODUCTION

A mechanical bonding between treated enamel surface and acrylic resin was made possible with the introduction of the acid etching technique by Buonocore.[1] Later, appearance of the first adhesive systems[2] and development of Bowen and Washinghton's[3] composite resins favored the improvement of esthetical adhesive materials.

In Pediatric Dentistry, pursue for esthetics, as in other Dentistry's areas, has increased, what has stimulated the development of adhesive systems capable of promoting an effective adhesion to enamel and dentin with a reduced number of steps during application.

The success of a restoration is based, upon other factors, on the sealing of the preparation margins. An effective adhesion to enamel and dentin reduces microleakage and bacterial penetration that may promote secondary caries and postoperative sensitivity.[4]

On total-etching technique, acid is applied simultaneously to enamel and dentin followed by a primer and a bonding resin. Adhesive systems that utilize total-etching are known as three-steps.[5] Due to time-consuming increased number of steps of these systems, adhesives that combine primer and bonding resin in just one step were developed.[5] To make this process even simpler new adhesives that do not need an acid etching step prior to its application were created, called self-etching.[5]

Self-etching systems partially demineralize the smear layer and subjacent dentin and promote the infiltration of the adhesive in these demineralized structures. These systems are less susceptible to manipulation variables once its application procedures are simpler and faster, what makes the process easier to be performed in children, avoiding contamination of the operatory field.[6,7] Results on the effectiveness of self-etching adhesive systems when compared to total-etching are contradictory.[6-10] Some show a similarity between these systems[6, 7, 10] while others suggest a superiority of the total-etching.[8,9]

Necessity of dentists (primarily Pediatric dentists) for simple and fast-application adhesives, allied to the importance of an effective bonding between tooth and restoration and doubts on the performance of different generations of adhesive systems on primary teeth justify the present study. The objectives of this study evaluated shear bond strength (SBS) of four adhesive systems to primary enamel and dentin; and verifying, after SBS testing, the failure mode occurred at the adhesive interface.

*Carla Miranda DDS,Msc, Graduate student, department of dental materials
**Luiz Henrique Maykot Prates DDS, MSc, PhD, Associate professor, department of dental materials
***Ricardo de Souza Vieira DDS, MSc, PhD, Associate professor, department of pediatric dentistry
****Maria Cristina Marino Calvo DDS, MSc, PhD, Associate professor, department of public health dentistry

Send all correspondence to: Carla Miranda, rua Joaquim Neves 114-A Pantano do Sul, Florianopolis CEP 88.067-120 SC, Brazil

Phone +55 (0) 48 32377 149

e-mail: ca_miranda@yahoo.com.br

METHODS AND MATERIALS

Teeth selection and storage

Sixty-two extracted sound primary human molars were selected and stored in 0.1% thymol in 0.9% saline solution (pH=7.0) for no longer than 6 months at room temperature. This research was approved by the Ethics Committee for Research with Human Beings of the Federal University of Santa Catarina (Process 283/04).

Shear bond strength (SBS) test

Specimen preparation

Teeth roots were wet-grounded with 100-grit silicon carbide (SiC) paper to the cement-enamel junction on a polishing machine (Panambra DP-10, Struers). Crowns were then sectioned on a mesial-distal direction with diamond discs (KG 7020, KG Sorensen). Each section was embedded in a polystyrene resin inside a 25 mm high and 20 mm diameter PVC tube, with buccal/lingual surface facing one of the extremities of the tube. After resin polymerization specimens were randomly divided into two groups:
1.) adhesion to dentin, and; 2.) adhesion to enamel.

For each specimen of the first group, dentin surface was exposed with 200-grit SiC paper grinding. Surface flattening and smear layer standardization were performed with 400- and 600-grit SiC paper. Grinding was carried out using a metallic support for the PVC tube dimensions on a polishing machine under water refrigeration in 4 different directions for 10 seconds (s) each.

For adhesion to enamel group, only a surface flattening was done. This procedure consisted of 400-grit wet-grinding of 0.4 mm followed by 600-grit wet-grinding of 0.1 mm. This 0.5 mm enamel removal was controlled by a digital precision caliper (727, Starett Tools).

After rinsing with distilled water and dried with oil-free compressed air, all grounded surfaces were delimited with adhesive paper leaving an uncovered area of 2 mm diameter. The 120 specimens were then randomly subdivided into four subgroups of 30 (15 dentin and 15 enamel each) according to the adhesive systems to be used (Scotchbond Multi-Purpose (3M ESPE), Single Bond (3M ESPE), Clearfil SE Bond (KURARAY) and Adper Prompt L-Pop (3M ESPE).

Bonding procedures

Application of adhesive systems was according to manufacturers instructions. Light-curing was performed with a halogen light curing unit (Curing Light 2500, 3M ESPE) with radiometer-controlled (Spring Health, Gnatus) light intensity of 550 mW/cm2.

A cylindrical bi-parted polytetrafluoroethylene (PTFE) 2 mm high mould with a 2 mm diameter orifice placed on the adhesive-treated surface was used for the composite resin (A2 shade) insertion. Fixation of the PVC cylinder and the PTFE mould were performed with a stainless steel device composed of two parts, an inferior one to hold the PVC cylinder and a superior one to hold the PTFE mould. The parts were attached to each other with screws so that the orifice of the mould perfectly coincided with the delimited adhesion area of the dental surface.

Composite resin was bulk inserted with an appropriate spatula (Composite 5, American Eagle) to fulfill the PTFE mould orifice and light cured for the time recommended by the manufacturers. The specimens' production was based on Cotto's method.[11]

Shear bond strength (SBS) test

After storage in distilled water at 37ºC for 24 hours (h) composite and adhesive flashes were removed with a scalpel blade. For SBS testing, conducted on a universal testing machine (4444, Instron), specimens were fixed on a stainless steel support so that a metallic apparatus with a semicircle notch of 3 mm diameter could apply a compressive force parallel to the dental surface at the base of the composite resin cylinder with a cross-head speed of 0,5 mm/min until fracture.

The bond strength values, expressed in megapascals (MPa), were obtained dividing the applied force (registered in Newtons) by the adhesive area (3.14 mm2). Data were subject to ANOVA and Tukey statistical tests ($p<0.05$).

Evaluation of the failure mode after SBS test

After 24 h of SBS testing, specimens were vacuum dried and sputter-coated with gold (SCD 005, Bal-tec) for evaluation of the failure mode on a scanning electron microscope (SEM) (XL-30, Philips) with 30X and 1000X magnification.

Failure modes classification was based on Sardella, Castro, Sanabe and Hebling's[9]:

1.) Cohesive: a) Composite resin: failure within composite resin; b) Adhesive: dental surface covered with an adhesive coating and/or dentinal tubules filled with adhesive; c) Dental structure: failure within enamel or dentin.

2.) Adhesive: failure between adhesive and dental structure, characterized by absence of adhesive covering dental surface or filling dentinal tubules.

3.) Mixed: failure had cohesive and adhesive patterns.

Analysis of the resin-dentin interface

Two teeth were prepared to obtain 4 dentin surfaces according to the previously described methodology. Each surface received one of the 4 tested adhesive systems and a 1 mm composite "restoration", according to manufacturers' instructions. Samples were sectioned perpendicularly to bonded surface in two halves. They were fixed in 2.5% glutaraldehyde in 0.2M sodium cacodylate buffer for 12 h at 4ºC. After fixation, samples were rinsed with 0.2M sodium cacodylate buffer for 1 h with two changes, followed by deionized water for 1 min. They were then dehydrated in ascendant grades of ethanol (25%, 50%, 75% for 20 min each; 95% for 30 min; and 100% for 1 h). After the final ethanol step samples were dried by immersion in hexamethyldisilazane (HMDS) for 10 min, placed on a filter paper and air-dried at room temperature. Samples were embedded in self-curing polystyrene resin and stored for 12 h. Epoxy casts were flattened on a polishing machine with SiC paper of decreasing abrasiveness (200-, 400-, 600-, 1200-grit) and polished with alumina pastes of 1 and 0.5mm for 1 and 5 min, respectively. Samples were ultrasonicated in 100% ethanol for 5 min, thoroughly dried, demineralized in 6M HCl for 30 sec, rinsed with deionized water and deproteinized in 2% NaOCl for 10 min, followed by rinsing with deionized water.

After drying, samples were sputter-coated with gold and observed by mean of scanning electron microscopy. Method was based upon Kaaden, Schmaltz and Powers.[12]

RESULTS

Shear bond strength

Mean and standard deviation (SD) values of SBS test are summarized on Table 1 and illustrated on Figure 1.

Failure mode evaluation

Failure modes for each adhesive system are presented in Table 2.

TABLE 1: Mean (MPa) SBS values of the tested adhesive systems on dentin and enamel.

Adhesive System	Surface			
	Dentin		Enamel	
	Mean (SD*)	CV*	Mean (SD*)	CV*
Scotchbond Multi-Purpose	17.29 (4.25) Ab	25%	27.89 (7.49) Aa	27%
Single Bond	18.2 (8.74) Aa	48%	23.92 (8.68) Aa	36%
Clearfil SE Bond	16.13 (7.14) Ab	44%	24.36 (6.69) Aa	27%
Adper Prompt L-Pop	6.04 (3.35) Bb	55%	25.96 (4.08) Aa	16%

Different capital letters in a same column and distinct small letters in the same line indicate statistical difference (ANOVA and Tukey, p<0.05). * SD =Standard deviation; CV =Coefficient of variation.

TABLE 2: Failure modes after SBS testing for all evaluated adhesive systems on dentin and enamel.

Adhesive System	Failure modes					
	Dentin			Enamel		
	Adhesive	Cohesive*	Mixed	Adhesive	Cohesive*	Mixed
Scotchbond Multi-Purpose	0	13	2	0	14	1
Single Bond	0	11	4	0	15	0
Clearfil SE Bond	0	15	0	0	15	0
Adper Prompt L-Pop	0	10	5	1	14	0

* Cohesive failure of the bonding agent.

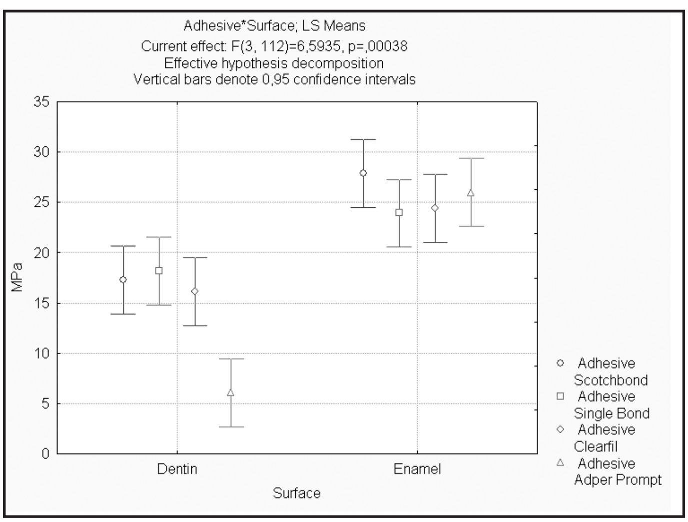

Figure 1: Graphic representation of SBS test results.

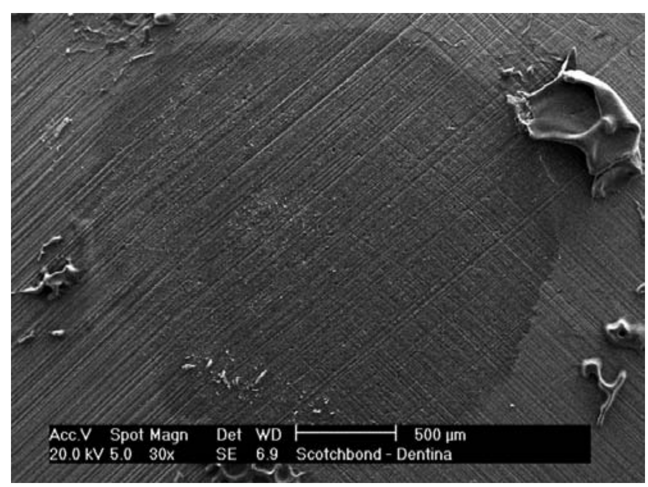

Figure 2: SEM micrograph of the dentin surface (30X magnification) with apparent adhesive failure.

Figures 2, 3 and 4 illustrate representative failures observed under SEM.

Resin-dentin interface analysis

Resin-dentin interface patterns observed under SEM are shown on figures 5 and 6.

DISCUSSION

Shear bond strength test was chosen for the present research for the following reasons: 1) it is a widely used and literature acclaimed test[6,7,10,13-16]; 2) does not stress specimens during sectioning procedures after bonding[7,17]; 3) easy execution and reproducibility.

Adhesive area used in this study (3.14 mm2) was smaller than other studies[14,15] because of the reduced dimensions of the primary tooth. Also, the smaller the adhesive area, lower is the probability of structural flaws within adhesive interface[17].

Overall SBS mean of the four evaluated adhesive systems to enamel was statistically higher than to dentin. However, this difference was not significant for SB probably because of the high coefficient of variation of this adhesive on enamel and dentin. Other studies have also verified superior bond strength values on enamel than on dentin[10,14]. This phenomenon is explained by differences of the structural composition of these substrates, being enamel almost totally composed of inorganic material and dentin characterized by

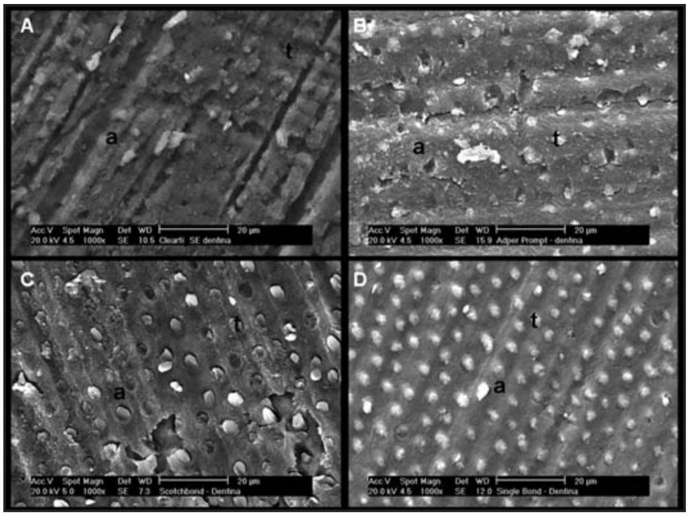

Figure 3: SEM micrographs of the dentin surface (1000X magnification). All images show tubular obliteration with resin tags (t) and an adhesive layer on the dentin surface (a), what indicates a cohesive failure mode of the adhesive. A - Clearfil SE Bond; B - Adper Prompt L-Pop; C - Scotchbond MP; D - Single Bond.

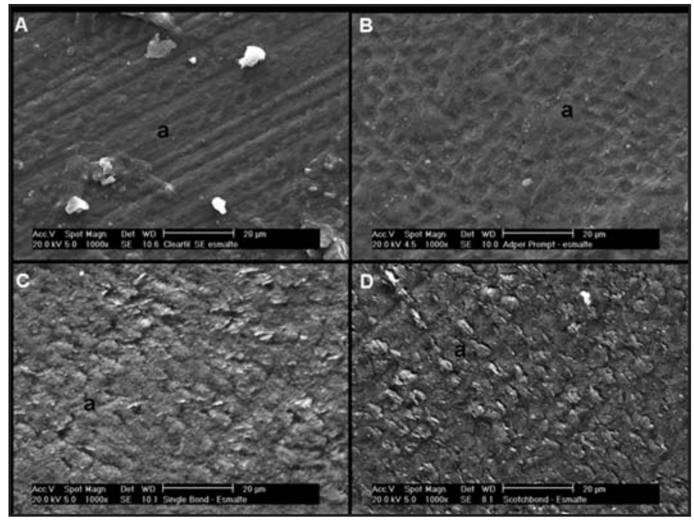

Figure 4: SEM micrographs of the enamel surface (1000X magnification). All images show adhesive remains (a) indicating a cohesive failure mode of the adhesive. A - Clearfil SE Bond; B - Adper Prompt L-Pop; C - Scotchbond MP; D - Single Bond.

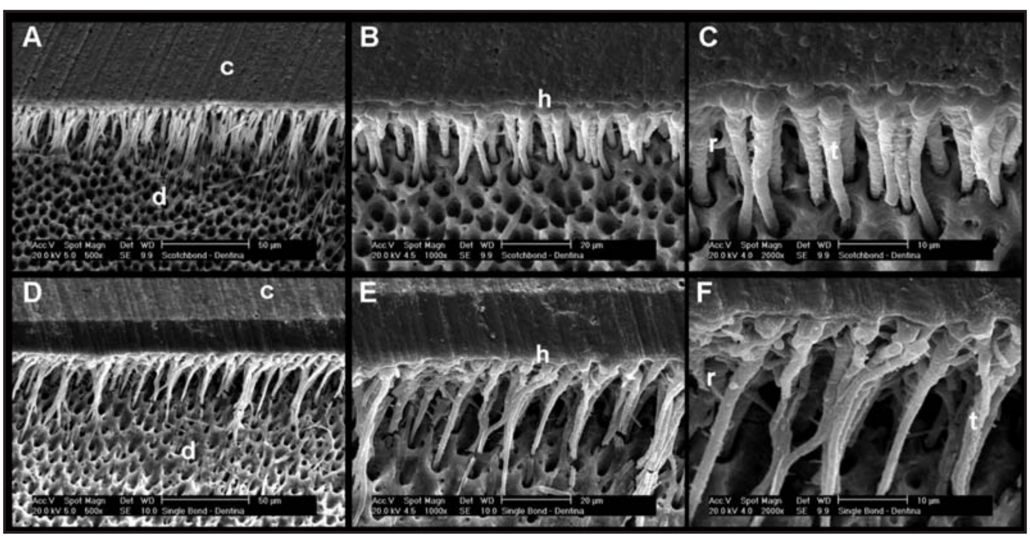

Figure 5: Resin-dentin interfaces SEM micrographs of the total-etching Scotchbond MP (A, B, C) and Single Bond (D, E, F) adhesive systems (A, D – 500X; B,E – 1000X; C, F- 2000X magnification). Thick hybrid layers are observed (h), as lateral ramifications (r) and numerous and long resin tags (t). (c-composite; d-dentin)

a high organic content and a tubular structure that is filled with odontoblastic processes and water. On dentin, as a result of cavity preparation, there is also formation of smear layer that acts as a barrier against an effective bonding of the adhesive system to the underlying dental structure.[5]

The four tested adhesive systems when applied to primary enamel presented statistically similar bond strengths what is corroborated by other studies.[13,18] However, this result disagrees with Perdigão and Geraldelli's[8] study that found higher SBS to enamel for total-etch than for self-ecthing adhesive systems. Nevertheless, these findings were on permanent enamel where some self-etching systems do not promote a satisfactory etching. On primary enamel, which seems to be more susceptible to demineralization[7], self-etching systems may promote good etching patterns, resulting in adequate bond strengths. In the present methodology, flattening of enamel was performed to reduce the accentuated axial surface convexity of primary teeth and to standardize an adhesive area. Some studies have shown that when enamel is not grounded there is the maintenance of the aprismatic layer, that is a superficial part of enamel less reactive to acid etching, what can explain an inadequate performance of self-etching adhesive systems, which may require a prior acid etching step or burgrounding of the enamel surface.[18,19]

On dentin APL, presented statistically lower SBS values than the other tested bonding agents. APL is considered a "strong" self-etching system (pH=0.4) resulting in an etching pattern similar to the one obtained with phosphoric acid[17]. On the other hand, on dentin, APL results were unsatisfactory when compared to a "mild" self-etching system as CSB10 (pH@2.0).[17] The organic solvent of APL is water, what may have influenced its performance once water, for its difficult removal, could remain within the adhesive interface.[17]

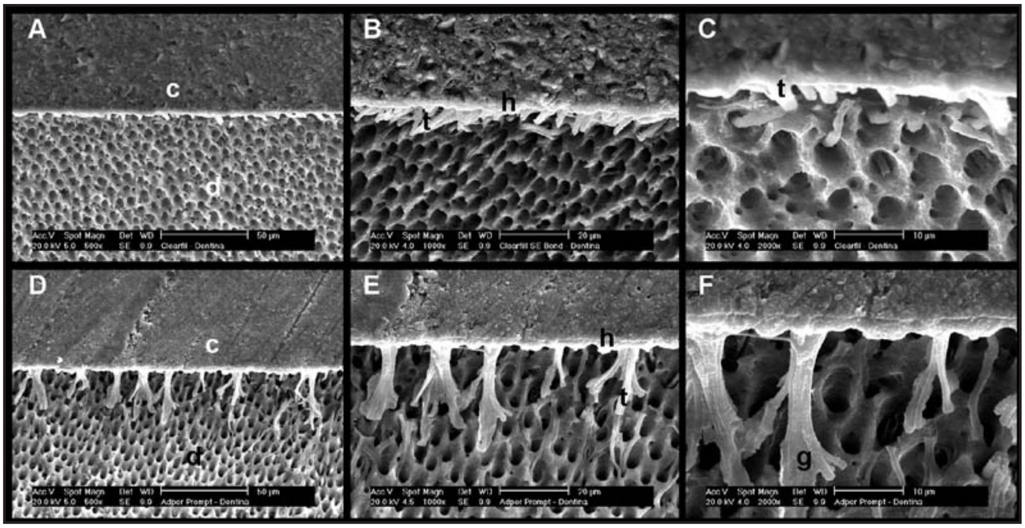

Figure 6: Resin-dentin interfaces SEM micrographs of the self-etching Clearfil SE Bond (A, B, C) and Adper Prompt L-Pop (D, E, F) adhesive systems (A, D – 500X; B,E – 1000X; C, F- 2000X magnification). Thin hybrid layers (h) and short resin tags (t) are observed. No lateral ramifications are visualized. Images of the APL adhesive (D,E, F) show grouped resin tags (g) in a smaller number than the other adhesive systems, what may indicate problems on the adhesive penetration. (c- composite; d- dentin)

CSB has as its solvent ethanol, that is easily removed from dentin surface by evaporation. CSB composition has also 10-MDP (10-methacryloyloxydecyl dihydrogen phosphate) which seems to be able to adhere to the remaining calcium of hydroxyapatite.[10,17] CSB is considered a filled adhesive while APL is unfilled, what turns the latter less resistant to mechanical loads.[5]

APL used in this study was presented as a single-dose three-blistered package which needs to be mixed prior to application on dental structure. This presentation makes bonding procedures easier, though effectiveness of liquid mixing may not be adequate, once part of them could be retained in curvatures of the blisters, avoiding a correct proportioning.

An acceptable marginal sealing on permanent dentin requires bond strength of around 17 MPa to compensate polymerization shrinkage stresses. When shrinkage stresses exceed bond strength a gap is formed between tooth and restoration.[20] Even though this value is not known for primary dentition, results of the present study are near of those required for an adequate bonding on permanent dentition for all adhesive systems, except for APL on dentin.

An analysis of the coefficient of variation of the tested adhesive systems (Table 1) demonstrates higher variations on dentin than on enamel. This is probably related to complexity of dentin substrate and its humidity.

Evaluation of the failure mode after SBS testing shows a predominance of cohesive failure, primarily of the adhesive, on both enamel and dentin surfaces, what also happened in other studies.[7,14,15] These SEM images mainly revealed failure within the bonding resin and dentinal tubules filled with resin tags or prismatic structure of enamel covered by an adhesive layer. Cohesive failures of bonding agent are related to high values of bond strength[15], predicting an effective bonding. It is also suggested that the expressive number of cohesive failures of the adhesive indicate a normal distribution of stresses during mechanical testing of bond strength.[17]

It is important to observe that failure mode evaluation should be done under SEM with high magnification and not only visually or with optical microscopes.[21] In the present study, lower magnifications (30X) revealed only an adhesive failure pattern (Figure 2). However, when high magnifications were used (1000X) (Figure 3C) a cohesive failure of the adhesive pattern was noticed, what is corroborated by El Kalla and García-Godoy´s study.[15]

Resin-dentin interface analysis for total-etch adhesive systems revealed a thick, well-defined hybrid layer with long resin tags and lateral tags. Both self-etching systems presented a thin hybrid layer, as observed by Senawongse, Harnirattisai, Shimada and Tagami.[6] APL showed a particularly low number of resin tags which seems to be grouped, what could be a sign of infiltration problems. A relationship between hybrid layer thickness and effective bonding was not established in literature[9], which is corroborated by the present study. Even though dentin-resin interface analysis was performed on a small number of samples with an illustrative character, a thin hybrid layer was observed for CSB despite its high bond strength values.

Considering the presented results and discussion it is important to stand out the necessity of more research evaluating primary dentition. These studies should be concentrated on dentin and evaluate the longevity of current adhesive systems.

CONCLUSIONS

1.) Overall SBS mean of the four evaluated adhesive systems to enamel was statistically higher than to dentin, even though this difference was not significant for subgroup SB.

2.) On enamel SBS was statistically similar for all four adhesives.

3.) On dentin SBS of APL was lower than SB, CSB and SMP, which were statistically similar between themselves.

4.) SEM analysis revealed a predominant cohesive failure mode (primarily of the bonding agent) for all adhesive systems on enamel and on dentin.

REFERENCES

1. Buonocore MG. A simple method of increasing the adhesion of acrylic filling materials to enamel surfaces. J Dent Res 34: 849-853, 1955.
2. Buonocore MG, Wileman W, Brudevold F. A report on a resin composition capable of bonding to human dentin surfaces. J Dent Res 35: 846-851, 1956.
3. Bowen RL, Washington DC. Properties of a silica-reinforced polymer for dental restorations. J Am Dent Ass 66: 57-64, 1963.
4. Bränström M, Nybörg H. Cavity treatment with a microbicidal fluoride solution: growth of bacteria and effect on the pulp. J Prosthet Dent 30: 303-310, 1973.
5. Van Meerbeek B, Perdigão J, Lambrechts P, Vanherle G. The clinical performance of adhesives. J Dent 26: 1-20, 1998.
6. Senawongse P, Harnirattisai C, Shimada Y, Tagami J. Effective bond strength of current adhesive systems on deciduous and permanent dentin. Oper Dent 29: 196-202, 2004.
7. Shimada Y, Senawongse P, Harnirattisai C, Burrow MF, Nakaoki Y, Tagami J. Bond strength of two adhesive systems to primary and permanent enamel. Oper Dent 27: 403-409, 2002.
8. Perdigão J, Geraldeli S. Bonding characteristics of self-etching adhesives to intact versus prepared enamel. J Esthet Restor Dent 15: 32-42, 2003.
9. Sardella TN, Castro FLA de, Sanabe ME, Hebling J. Shortening of primary dentin etching time and its implication on bond strength. J Dent 33: 355-362, 2005.
10. Atash R, Van Den Abbeele A. Bond strengths of eight contemporary adhesives to enamel and dentine: an in Vitro study on bovine primary teeth. Int J Paediatr Dent 15: 264-273, 2005.
11. Cotto, RAC. Influência do cimento de óxido de zinco e eugenol para restauração temporária na resistência de união de sistemas adesivos. [Dissertation]. Porto Alegre (RS): Pontifica Universidade Católica do Rio Grande do Sul; 2002. 111 p.
12. Kaaden C, Schmalz G, Powers JM. Morphological characterization of the resin-dentin interface in primary teeth. Clin Oral Invest 7: 235-240, 2003.
13. Shimada Y, Iwamoto N, Kawashima M, Burrow MF, Tagami J. Shear bond strength of current adhesive systems to enamel, dentin and dentin-enamel junction region. Oper Dent 28: 585-590, 2003.
14. Fritz U, García-Godoy F, Finger WJ. Enamel and dentin bond strength and bonding mechanism to dentin of Gluma CPS to primary teeth. J Dent Child 64: 32-38, 1997.
15. El Kalla IH, García-Godoy F. Bond strength and interfacial micromorphology of four adhesive systems in primary and permanent molars. J Dent Child 65: 169-176, 1998.
16. Al-Salehi SK, Burke FJT. Methods used in dentin bonding tests: an analysis of 50 investigations on bond strength. Quintessence Int 28: 717-723, 1997.
17. Van Meerbeek B, De Munck J, Yoshida Y, Inoue S, Vargas M, Vijay P, Van Landuyt K, Lambrechts P, Vanherle G. Adhesion to enamel and dentin: Current status and future challenges. Oper Dent 28: 215-235, 2003.
18. Kanemura N, Sano H, Tagami J. Tensile bond strength to and SEM evaluation of ground and intact enamel surfaces. J Dent 27: 523-530, 1999.
19. Meola MT, Papaccio G. A scanning electrón microscope study of the effect of etching time and mechanical pre-treatment on the pattern of acid etching on the enamel of primary teeth. Int Dent J 36: 49-53, 1986.
20. Davidson CL, Gee AJ de, Feilzer A. The competition between the com posite-dentin bond strength and the polymerization contraction stress. J Dent Res 63: 1396-1399, 1984.
21. Pashley DH, Sano H, Ciucchi B, Yoshiyama M, Carvalho RM. Adhesion testing of dentin bonding agents: a review. Dent Mater 11: 117-125, 1995.
22. Wang Y, Spencer P. Physicochemical interactions at the interfaces between self-etch adhesive systems and dentine. J Dent 32: 567-579, 2004.