CORRELATION OF THE HYBRID LAYER THICKNESS AND RESIN TAGS LENGTH WITH THE BOND STRENGTH OF A SELF-ETCHING ADHESIVE SYSTEM

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ABSTRACT

The objective of this study was to measure the thickness of the hybrid layer (HLT), length of resin tags (RTL) and bond strength (BS) in the same teeth, using a self-etching adhesive system Adper Prompt L Pop to intact dentin and to analyze the correlation between HLT and RTL and their BS. Ten human molars were used for the restorative procedures and each restored tooth was sectioned in mesio-distal direction. One section was submitted to light microscopy analysis of HLT and RTL (400×). Another section was prepared and submitted to the microtensile bond test (0.5 mm/min). The fractured surfaces were analyzed using scanning electron microscopy to determine the failure pattern. Correlation between HLT and RTL with the BS data was analyzed by linear regression. The mean values of HLT, RTL and BS were 3.36 µm, 12.97 µm and 14.10 MPa, respectively. No significant relationship between BS and HLT (R^2= 0.011, p>0.05) and between BS and RTL (R^2= 0.038) was observed. The results suggested that there was no significant correlation between the HLT and RTL with the BS of the self-etching adhesive to dentin.

Key words: dentin, dentin-bonding agents, tensile strength, microscopy.

CORRELAÇÃO DA ESPESSURA DA CAMADA HÍBRIDA E DO COMPRIMENTO DOS PROLONGAMENTOS RESINOSOS COM A RESISTÊNCIA DE UNIÃO DE UM ADESIVO AUTOCONDICIONANTE.

RESUMO

O objetivo dessa pesquisa foi mensurar a espessura da camada híbrida de adesão (CH), o comprimento dos prolongamentos resinosos (Tags) e a resistência de união (RU) em um mesmo espécime e analisar a correlação entre esses fatores, usando o adesivo autocondicionante Adper Prompt L Pop em dentina hígida. Dez molares humanos foram utilizados e após a realização dos procedimentos restauradores, de acordo com os fabricantes, cada espécime foi cortado ao meio no sentido mésio/distal. Em uma hemi-secção dental os espécimes foram descalcificados para análise e mensuração dos tags e da camada híbrida de adesão em microscopia óptica comum (AXIOPHOT, 400X). Na outra hemi-secção, foi realizado o teste de microtração em uma velocidade de 0,5 mm/min até sua ruptura. A superfície fraturada foi mensurada e classificada de acordo com o tipo de fratura observada em microscopia eletrônica de varredura. Os valores obtidos para os fatores em análise, correspondentes a cada espécime foram submetidos a um teste de correlação. As médias correspondentes a CH, Tags e RU foram 3,36µm, 12,97 µm 14,10 MPa, respectivamente. Não foi observado correlação entre a CH e RU (R^2= 0,011, p>0,05) e entre os Tags e RU (R^2= 0,038). Diante dos resultados, observamos não haver correlação entre a camada híbrida e a resistência à tração, assim como entre os tags e a resistência à tração do sistema adesivo autocondicionante empregado.

Palavras chaves: dentina, adesivos dentinários, força de união, microscopia óptica comum.

INTRODUCTION

Adhesive systems are indispensable in current dental practice. The efficiency of bonding to dentin depends on micromechanical retention promoted by resin infiltration in partially demineralized dentin, leading to the formation of the hybrid layer and tags[1]. To fulfill these requirements, there are two strategies: the etch-&-rinse and self-etch approaches[2].

Self-etching adhesives have been developed in an attempt to reduce technique sensitivity and simplify the clinical steps of the adhesive technique. They do not require previous acid etching and simultaneously provide enamel and dentin surface demineralization, followed by infiltration of resin monomers[3].

Most of information in the literature on adhesive systems has been obtained by electron microscopy studies, which provide images of small resin-dentin interface areas. However, little consistent information is available about the performance and the

Table 1: Materials employed in this study (components, manufacturers).

Material	Composition
Adper Prompt L Pop 3M/ESPE, St Paul, MN, US	(Liquid 1 (red compartment) – methacrylate esters derived from phosphoric acid, Bis-GMA, camphorquinone initiators, stabilizers; Liquid 2 (yellow compartment) – water, HEMA, polyalkenoic acid, stabilizers, methacrylate ester derived from phosphoric acid, fluoride compounds) (3M/ESPE, St Paul, MN, USA)
Z 250	UDMA: urethane dimethacrylate, Bis-EMA: Bisphenol A – polyethylene glycol dieter, dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; inorganic fillers

ability of these systems in large areas, as reported by some authors[4,5,6].

Conversely, Sano et al. developed the microtensile bond test, which evaluates the bond strength in small bonded areas[7]. Compared to conventional tests, this method has two important advantages: homogeneous stress distribution at the bonded interface and low incidence of cohesive fracture in the substrate or in restorative composite, both of which contribute to the measurement of actual bond strength[8,9].

The literature has described the presence of the hybrid layer and resin tags[6,10,11] and has reported several results of microtensile bond strength to dentin for self-etching adhesives[12,13]. However, few studies have evaluated the correlation between the length of resin tags and hybrid layer thickness with bond strength to dentin[14,15], mainly evaluated in the same specimen. The objective of this study was to evaluate the bond strength, measure the hybrid layer thickness and the length of resin tags of a self-etching adhesive to dentin and correlate the bond strength with the hybrid layer thickness and the length of resin tags in the same tooth. The null hypothesis tested was that bond strength is not influenced by the hybrid layer thickness and the length of resin tags.

MATERIAL AND METHODS

Specimen Preparation and Bonding Procedures

Ten intact third human molars, which were stored in distilled water, were used in this study (up to 6 months after extraction). The study was revised and approved by the Institutional Review Board (Araçatuba—UNESP). The single-step self-etching adhesive system Adper Prompt L Pop (3M ESPE, Seefeld, Germany), and the composite resin (Filtek™ Z250, 3M ESPE, St. Paul, MN, USA) (Table 1) were used.

The occlusal enamel was removed with a diamond disc (IsoMet Diamond Wafering Blade, Buehler Ltd, Lake Bluff, IL, USA) under constant water irrigation. The occlusal surface was abraded with silicon-carbide sandpaper grit 320 under water irrigation on a polishing machine (Fortel Ltda, São Paulo, SP, Brazil) to expose the middle-depth dentin. A standardized smear layer was then created with silicon-carbide sandpaper grit 600, under continuous irrigation for 30 seconds.

The adhesive system Adper Prompt L Pop was applied on the dentin surface following the manufacturer's instructions and was light cured for 20 seconds (Ultralux Lens, Dabi Atlante, Ribeirão Preto, SP, Brazil) at an intensity of 450mW/cm^2. A Filtek Z250 composite resin block (shade A2) measuring nearly 4 mm in height was incrementally built-up on dentin surfaces and each increment was light-cured for 40 seconds. The bonding procedures were performed in controlled environmental conditions at 22°C under 45% to 55% of humidity. Each restored tooth was sectioned mesio-distally with a diamond disc (IsoMet Diamond Wafering Blade, Buehler Ltd, Lake Bluff, IL, USA) under constant irrigation on a sectioning machine ISOMET 2000 (Buehler, Lake Bluff, IL, USA) to obtain two (buccal and lingual) hemi-samples.

Light Microscopy Analysis

The hemi-samples that were used for optical microscopy were decalcified in 50% formic acid and 20% sodium citrate water solution, which was changed after 5 days. Decalcification of each specimen was monitored radiographically[6,11]. Complete decalcification was achieved after 3 months. This process completely removed the dental enamel, leaving only the demineralized dentin tissue, which was the object of evaluation in the present study. After decalcification, the restorations were carefully removed and embedded in paraffin. Then, the

decalcified hemi-samples were sectioned (ISOMET 2000 - Buehler, Lake Bluff, IL, USA) longitudinally through their crowns at 6 µm and mounted on glass slides. Fifteen slides of each hemi-sample, containing approximately six sections each, were selected by systematic sampling, with an interval proportional to the number of sections obtained for each hemi-sample[6,11]. These sections were stained with the Brown and Brenn stain[16] and the best histological sections, showing the best stained hybrid layer and tags were analyzed on a light microscope (Axiophot, Zeiss DSM-940 A, Carl Zeiss MicroImaging Inc, Thornwood, NY, USA) at 400X magnification, with a micrometric 40/075 ocular lens (or eyepiece) (Fig. 1). The hybrid layer and resin tags of each section were measured by a single, calibrated examiner over the entire extension of the histological section. Three measurements were recorded per section for hybrid layer thickness and the length of resin tags. The mean of the three measurements was recorded as the thickness of the hybrid layer and the length of the resin tags. Thus, fifteen mean values were obtained for each hemi-sample, for both the hybrid layer and the resin tags.

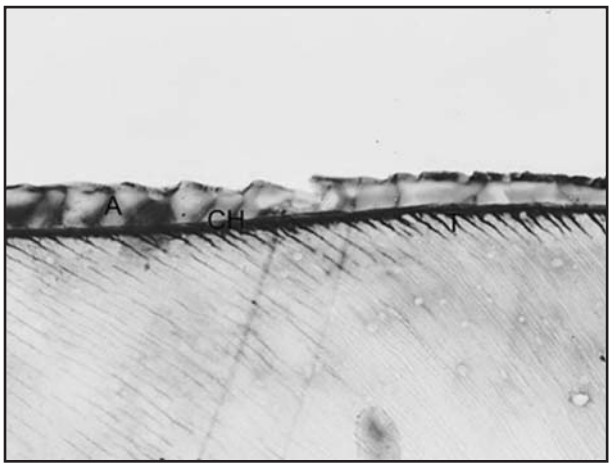

Fig. 1: Light microscope image (400X magnification), revealing hybrid layer and resin tag formation. (A – Adhesive; CH – hybrid layer and T – resin tags.)

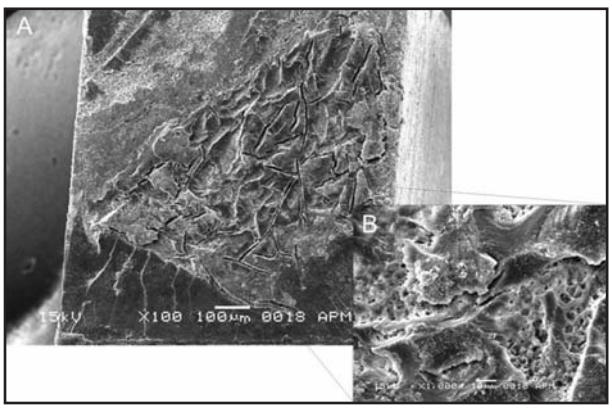

Fig. 2: SEM photomicrograph, revealing adhesive fracture pattern. A) 100X magnification. B) 1000X magnification revealing many pores of the adhesive layer.

Microtensile Bonding Test

The other hemi-samples of restored teeth were used for the microtensile bond strength test. The hemi-sample teeth were serially sectioned vertically into several 1 mm thick slabs with a diamond disc. Each slab was further sectioned to produce several bonded sticks of approximately 1.0 mm^2. Each bonded stick was fixed to the grips of a testing device (Instron model 4411 Instron Inc., Canton, MA, USA) with cyanoacrylate glue (Super Bonder - Henkel Ltda., Itapevi, Sao Paulo, Brazil) and tested under tension at 0.5 mm/min crosshead speed until failure. After testing, the specimens were carefully removed from the fixtures with a scalpel blade and the cross-sectional area at the site of fracture was measured to the nearest 0.01 mm with a digital caliper (Digimess, Shinko Precision Gaging, LTD, China) to calculate bond strength that was expressed in MPa.

The dentin side of failed specimens was sputter-coated with gold (Balzers SCD 050, Balzers Union, Balzers, Liechtenstein) and observed under a SEM (JSM 5600 LV, Jeol Inc., Peabody, MA, USA). Photomicrographs of a representative area of the surface were taken at 100X and 1000X magnification (Fig. 2). The fracture patterns were classified as adhesive, cohesive in dentin, cohesive in composite, or mixed if more than one structure was involved in the fracture.

Data treatment

Individual bond strength values (n=10) were correlated with hybrid layer thickness and length of resin tags and analyzed by linear regression. Statistical significance was set at a = 0.05.

RESULTS

The values of hybrid layer thickness, resin tag length and bond strength are presented in Table 2. The mean values of hybrid layer thickness, resin tag length and bond strength were 3.36 µm, 12.97 µm and 14.10 MPa, respectively.

Table 2: Values of hybrid layer thickness, resin tag length and bond strength.

Specimen	Hybrid layer thickness	Resin tag length	Bond strength
1	3.39 µm	14.06 µm	16.4 MPa
2	3.83 µm	13.00 µm	10.34 MPa
3	3.11 µm	13.17 µm	10.28 MPa
4	3.94 µm	15.56 µm	13.91 MPa
5	3.06 µm	13.67 µm	16.55 MPa
6	3.22 µm	14.28 µm	19.21 MPa
7	3.94 µm	8.61 µm	21.13 MPa
8	3.00 µm	12.94 µm	10.07 MPa
9	2.33 µm	11.56 µm	13.69 MPa
10	3.72 µm	11.56 µm	9.51 MPa

The self-etching adhesive Adper Prompt L-Pop exhibited a high percentage of adhesive fractures (69%), followed by cohesive fractures in resin (17%) and mixed fractures (14%). No dentin fracture was observed.

There was no significant correlation between bond strength and hybrid layer thickness (R^2= 0.011, p>0.05) and between bond strength and resin tag length (R^2= 0.038, p>0.05).

DISCUSSION

This study evaluated the ability of penetration and bond strength of the adhesive material Adper Prompt L-Pop to intact dentin tissue. The light microscopy analysis allowed the assessment and measurement of the thickness of the hybrid layer and length of resin tags, in an extensive dentin area within the same tooth (Fig. 1), thus yielding consistent information, as reported by other authors[5,6,11]. These resin structures are intensely stained by the Brown & Brenn method[16], allowing adequate microscopic observation of the structures[11].

The mean value of bond strength of Adper Prompt L-Pop self-etching adhesive to dentin was 14.10 MPa, which can be considered a low value compared to conventional etch&rinse systems and self-priming adhesives[17,18]. This mean value corroborates other studies that showed similar values[11,19-22]. The Gregoire & Millas study (2005)[23] reported a lower mean value than that reported herein. Regarding the resin tag length, this study showed a mean value similar to that reported by Sundfeld et al. (2005)[4] and Lohbauer et al. (2007)[18]. The hybrid layer has been described as thicker than for one- or two-step self-etching systems[9,21,23], which can form a thin hybrid layer of one or two µm and short resin tags[24].

Adper Prompt L-Pop self- etching adhesive is considered a strong self-etch adhesive with a very low pH (0.35)[23] and high concentration of hydrophilic monomers. The adhesive hydrophilicity results in increased water sorption, decreasing water stability. Moreover, the lack of hydrophobic components at resin-dentin interfaces may be responsible for the low values of bond strength[25,26]. The simplification of bonding procedures has resulted in loss of bonding effectiveness due to the more hydrophilic nature of this adhesive that forms a hybrid layer that is more permeable to water[27]. Clinically, it is not easy to evaporate the water of these adhesive solutions after applying on the dentin surface. The water is necessary to provide the medium for ionization and action of acidic resin monomers. However, the residual water can impair the polymerization of this adhesive and the mechanical properties of the hybrid layer[28,29]. Thus, the low bond strength and the high incidence of adhesive failures found in this study are related to the hybridization process and the chemical characteristics of the adhesive.

A study published by Anchieta et al., 2008[17] showed a significant relationship between bond strength of conventional 3-step etch&rinse adhesive and hybrid layer thickness. However, in this study this correlation was not observed and the null hypothesis was accepted. The lack of correlation observed between the length of resin tags and the bond strength for the self-etching adhesive system Adper Prompt L Pop can be explained by the report of Wang & Spencer, in 2002[30]. These authors stated that the application of a self-etching adhesive on dentin promotes deeper migration of molecules with lower molecular weight such as hydrophilic monomers (HEMA). Therefore, most of the tags are formed by monomers with low molecular weight, which are weakly cured, reducing their contribution to the bond strength[17,28].

CONCLUSION

Within the limits of these experiments it can be concluded that the bond strength of the one-step self-etching adhesive to dentin is not dependent on the hybrid layer thickness and length of resin tags.

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