

Analysis of the microtensile bond strength to enamel of two adhesive systems polymerized by halogen light or LED

Análise da microtração em esmalte de dois sistemas adesivos ativados por LED ou por luz halógena

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ABSTRACT: With the development of composite resin polymerization techniques, LEDs were introduced to the market place. Based on the studied literature, the aim of this study was to evaluate, through microtensile testing, the bond strength to enamel of a composite resin associated to a conventional (multi-bottle) adhesive system or to a self etching primer adhesive system, polymerized by halogen light or LED (light emitting diode). Bovine teeth were divided into 4 groups with 10 teeth each ($n = 10$). Then the adhesives Scotchbond Multipurpose Plus (3M-ESPE) and Clearfil SE Bond (Kuraray) were applied following the manufacturers' instructions. Both systems were polymerized for 10 s by halogen light (Degulux Soft Start – Degussa Hulls) set at 550 mW/cm^2 or by LED (Kerr Demetron) set at 600 mW/cm^2 . The composite resin Filtek Z-250 was applied in four 1 mm increments with the aid of a square, condensation silicon, 5 mm x 5 mm matrix, and polymerized by either of light sources for 40 s. Scotchbond Multipurpose Plus polymerized by halogen light presented the highest bond strength values ($39.69 \pm 7.07 \text{ MPa}$), and the other groups did not present statistically significant differences: Scotchbond Multipurpose Plus polymerized by LED ($22.28 \pm 2.63 \text{ MPa}$), Clearfil SE Bond polymerized by halogen light ($27.82 \pm 2.65 \text{ MPa}$) and by LED ($22.89 \pm 5.09 \text{ MPa}$).

DESCRIPTORS: Adhesives; Dental bonding; Polymerization; Light-emitting diode (LED).

RESUMO: Com a grande busca pelo desenvolvimento das técnicas de polimerização das resinas compostas e dos sistemas adesivos, foram introduzidos no mercado os LEDs. Com base na literatura estudada, neste trabalho é avaliada a força de união da resina composta ao esmalte associado a um sistema adesivo convencional e a um sistema autocondicionante polimerizados por luz halógena e LED, por meio do teste de microtração. Os dentes bovinos foram divididos em 4 grupos com 10 dentes em cada um ($n = 10$), para aplicação do adesivo Scotchbond Multipurpose Plus (3M-ESPE) e do Clearfil SE Bond (Kuraray) conforme orientação do fabricante, ambos polimerizados por 10 s tanto pela luz halógena (Degulux Soft Start – Degussa Hulls) com potência de 550 mW/cm^2 como pelo LED (Kerr Demetron) com potência de 600 mW/cm^2 . A resina composta Filtek Z-250 foi aplicada em 4 incrementos de 1 mm com auxílio de uma matriz de silicóna de condensação de formato quadrado com dimensões de 5 mm x 5 mm; sendo polimerizada tanto pela luz halógena como pelo LED por 40 s. O adesivo Scotchbond Multipurpose Plus polimerizado com luz halógena apresentou os maiores valores de adesão ($39,69 \pm 7,07 \text{ MPa}$) e os outros grupos não apresentaram diferença estatisticamente significante entre si: Scotchbond Multipurpose Plus polimerizado por LED ($22,28 \pm 2,63 \text{ MPa}$), Clearfil SE Bond polimerizado por luz halógena ($27,82 \pm 2,65 \text{ MPa}$) e por LED ($22,89 \pm 5,09 \text{ MPa}$).

DESCRITORES: Adesivos; Colagem dentária; Polimerização; Light-emitting diode (LED).

INTRODUCTION

Dentistry is developing with the purpose of contributing to a better clinical performance of dental materials. New technologies have been introduced, and among them is the light emitting diode technology (LED). LEDs use gallium nitride semiconductors and, when subjected to an electric current, they produce blue light.

This polymerization system emits a light with a wavelength between 450 and 490 nm, with a peak of 470 nm^{15} , which activates the camphoroquinone. The photo-initiator phenyl propadione (PPD) has an absorption peak of 420 nm^{18} , which means that LEDs are not able to start polymerization of composite resins and adhesive systems that

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contain this photo-initiator.

LEDs have some advantages over halogen light: because of the smaller energy necessity, they promote little heat¹⁴ and have a longer lifetime³. In addition, fans are not needed to cool the curing unit³.

A study suggested that LEDs should be applied for enough time to promote composite resin monomer conversion⁵. Some studies have related a worse performance of LEDs when compared to that of halogen light^{1,12,14}.

Therefore there is the necessity of more studies to evaluate LEDs' performance. Furthermore, it is not certain that LEDs are really efficient when used for the polymerization of adhesive systems, and of darker or microfilled composite resins.

Another aspect to be considered is the efficacy of the bond strength to enamel of self-etching primers. Self-etching primers represent a new concept in adhesion because the acidic primer promotes simultaneously surface etching and its penetration into enamel or dentin. Recently, the effect of self-etching primers on enamel has been studied more frequently because it produces an etch pattern different from that obtained with multi-bottle adhesives. It was observed that the etching pattern of self-etching primers had a smaller quantity of microporosities⁸. A lower degree of acid etching was also observed²⁵, although satisfactory bond strength was obtained²⁷.

In spite of the multiple steps required by multi-bottle adhesive systems, they are considered effective. Multi-bottle adhesive systems (Scotchbond Multipurpose Plus) have presented greater bond strengths². This result is due to good monomer impregnation in dentin and enamel, 2 to 4 μm of hybrid layer, and a uniform adhesive layer (30 μm)².

Based on a review of the literature, the aim of this study was to evaluate LED efficacy on the polymerization of different adhesive systems and also to compare the bond strength to bovine enamel of a multi-bottle adhesive system (Scotchbond Multipurpose Plus) and that of a self-etching primer (Clearfil SE Bond).

MATERIAL AND METHODS

Forty freshly extracted Bovine teeth were separated from their root portions to be used in this study, as some authors have confirmed their applicability as a substitute for human enamel for adhesion testing¹⁷. After that, their buccal surfaces were ground in a water-cooled mechanical

grinder (Politriz-Buehler, Boston, MA, USA), using a 600 grit Al_2O_3 abrasive paper to obtain flat, standardized enamel surfaces. The teeth were then stored in distilled water.

The teeth were divided into 4 groups with 10 teeth each ($n = 10$) as follows:

- **Group 1** – Scotchbond Multipurpose Plus (3M ESPE, St. Paul, MN, USA), polymerized by halogen light.
- **Group 2** – Scotchbond Multipurpose Plus, polymerized by LED.
- **Group 3** – Self-etching primer Clearfil SE Bond (Kuraray Co., Osaka, Japan), polymerized by halogen light.
- **Group 4** – Self-etching primer Clearfil SE Bond, polymerized by LED.

The adhesive systems were applied following the manufacturers' instructions and were light-cured for 10 seconds with a light curing unit (Degulux Soft Start, Degussa Hills, Hanau, Germany) set at 550 mW/cm^2 , or with a LED unit (Kerr Demetron, Danbury, CT, USA) set at 600 mW/cm^2 . After that, the Filtek Z-250 (3M ESPE, St. Paul, MN, USA) composite resin was inserted in 4 increments of 1 mm each and light-cured for 40 seconds. A silicon-based impression material (Optosil, Heraeus Kulzer GmbH & Co., KG, Germany) was used as a matrix with dimensions of 5 mm x 5 mm.

The choice of microtensile bond testing was based on studies confirming that higher bond strength values are obtained in smaller areas^{22,23}.

In order to obtain the specimens, the teeth were mounted on an acrylic resin base. Composite resin and tooth structure were cut with a diamond disc in the cervico-occlusal and mesiodistal directions. The specimens thus prepared had a rectangular cross-sectional area of approximately 1 mm^2 .⁴ For cutting, the Lab Cut 1010 (Exttec corp. – Enfield, CT, USA) machine was used with a diamond disc (Exttec corp. – Enfield, CT, USA), set at 250 rpm of speed for the mesiodistal direction and at 125 rpm for the cervico-occlusal direction. Only the 4 central specimens of each tooth were used for the microtensile bond testing.

The composite resin increments were inserted so as to maintain the proportion between the lengths of tooth structure and composite resin. A pattern was thus created to avoid any influence that could be caused by different dimensions of the 2 extremities during the stress applied in the microtensile test.

The specimens were then fixed at either side with cyanoacrylate adhesive (Super Bonder Gel,

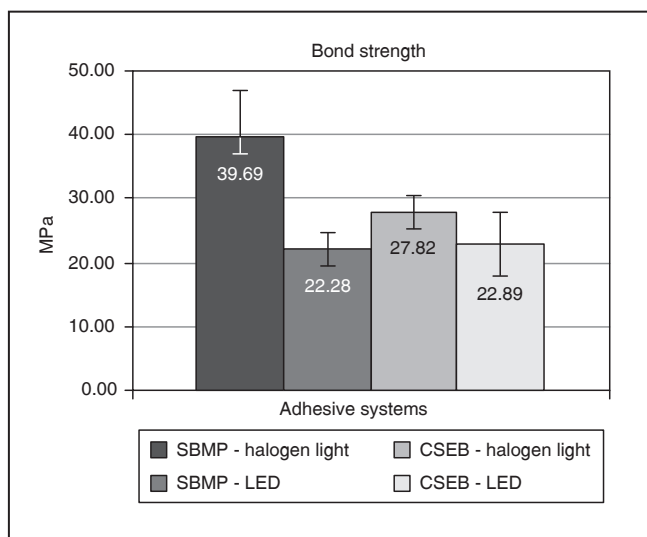
Loctite, São Paulo, SP, Brazil) in the Geraldeli device²¹, which was used for the microtensile bond test. Hence, the area of adhesion was placed perpendicular to the direction of the applied stress. The specimen device was mounted in the universal testing machine Mini Instron model 4442 (Instron – Canton, CT, USA) set at a speed of 0.5 mm/min.

Three types of fracture were obtained: in the adhesive junction, in the enamel area and in the composite resin area; mixed fractures were also observed. However, only the fractures in the adhesive junction were measured with a caliper and, after that, the bond strength values were transformed to MPa. Using this methodology, the bond strength values were obtained according to the cross-sectional specimen areas. The values were submitted to statistical analysis of variance (ANOVA) and to Tukey's test.

The original results consist of 160 bond strength values (in MPa) related to 40 teeth, with 4 specimens per tooth. Furthermore, only specimens with adhesive fracture were used for the statistics. An average of these values was then calculated, resulting in 40 values, 10 for each group.

RESULTS

The values submitted to statistical analysis of variance (ANOVA) presented statistical significance for 2 factors: source (F = 55.55) and adhesive (F = 14.12), and for the relation source *versus* adhesive (F = 17.35). With the relation averages,



GRAPH 1 - Microtensile bond strength to enamel values of two adhesive systems polymerized by halogen light and LED.

Tukey's test was done at the 5% level of significance. Scotchbond Multipurpose Plus polymerized with halogen light presented higher bond strength values and there was no statistically significant difference between the other groups.

The averages and standard deviations for the 4 groups are presented in Graph 1 and Table 1.

DISCUSSION

In literature, some differences between polymerization with halogen light and with LED are reported.

In a comparison of LED *versus* halogen light and another source of polymerization, it was concluded that it is necessary to consider curing unit power, LED number and exposure time. Only powerful LEDs can be compared to halogen light¹.

Some studies have described LED results *versus* halogen light results. The composite resin polymerization depth of a curing unit with 27 LEDs set at 350 mW/cm² was 20% smaller than that of halogen light¹². Exposure time needed by LEDs for an adequate composite resin polymerization was longer than that required by halogen light¹⁴. The composite resin mechanical properties obtained after polymerization with LEDs set at 300 mW/cm² were inferior or similar to those obtained with halogen light, however the LED results fulfilled the requirements set by the International Standards Organization (ISO)¹. The poorer performance (inferior mechanical properties) of LEDs can be explained by the use of a unit with 7 LEDs with only 150 mW/cm².⁷ A curing unit with 61 LEDs promoted a greater depth of polymerization of composite resins than that of halogen light⁹.

In another study, only the Elipar FreeLight LED (3M-ESPE) set at 400 mW/cm² achieved adequate polymerization of superficial and deeper layers of composite resin specimens²⁶. This means that this LED promoted the same hardness val-

TABLE 1 - Microtensile bond strength to enamel values of two adhesive systems polymerized by halogen light and LED.

Groups	Mean ± sd
SBMP-halogen light	39.69 ± 7.07 MPa
SBMP-LED	22.28 ± 2.63 MPa
CSEB-halogen light	27.82 ± 2.65 MPa
CSEB - LED	22.89 ± 5.09 MPa

SBMP: Scotchbond Multipurpose Plus. CSEB: Clearfil SE Bond.

ues as those of halogen light²⁶. The hardness values – and, therefore, the effectiveness of composite resin polymerization – obtained with the GC e-light LED (GC), set at 600 mW/cm² were lower than those produced by halogen light at 800 mW/cm².²⁶ Therefore, effectiveness of LED curing lights depends on the kind of curing unit used²⁶.

It was also observed that only a more powerful LED unit (320 mW/cm²) obtained hardness values of the Filtek Z-250 composite resin similar to those of halogen light (800 mW/cm²)¹¹. A less powerful LED unit (160 mW/cm²) used in the same study needed more exposure time to obtain results similar to those of halogen light¹¹.

When comparing a LED unit at 300 mW/cm² to a conventional light set at 455 mW/cm², one study related that the depths of polymerization of flowable, microfill and hybrid composite resins obtained with LED were greater than those obtained with halogen light¹⁵.

A LED curing unit with 64% of halogen light irradiation produced a satisfactory depth of polymerization of composite resins¹⁵. Moreover, LED can be useful in clinical practice because its performance is not significantly reduced with the reduction of exposure time, contrasting with the behavior of halogen light¹⁵.

All these studies permitted to conclude that the use of reliable and powerful LED light-curing units is advisable in order to produce effective polymerization and bond strength results similar to those of halogen light.

Some studies have correlated depth of polymerization and monomer degree of conversion^{12,15}. An adequate polymerization of adhesive systems and composite resins is a guarantee of good clinical results. Moreover, higher molecular weight monomers as Bis-GMA or urethane dimethacrylate (UDMA) always have residual non-reacting C=C, mainly because of limitations in the mobility of reactive groups caused by the rapid formation of a cross-linked polymeric network⁶. Hence, a powerful and effective light-curing unit is necessary for an adequate polymerization.

Based on the related literature, it can be assumed that the inferior performance of the LED curing unit (LED Kerr Demetron) used in this study to polymerize the Scotchbond Multipurpose Plus adhesive can be explained because this LED unit promoted a lower degree of polymerization when compared to the halogen light Degulux. Therefore, the Kerr Demetron LED unit set at 600 mW/cm², when applied for 10 s, is not equivalent to the Degulux Soft Start halogen light unit set at 550 mW/

cm² applied for the same 10 s. It is advisable to use the LED curing unit used in this study for a longer period of time.

The similar bond strength values obtained in this study when Clearfil SE Bond was polymerized with halogen and with LED can be explained by the unreliable performance of this adhesive in enamel. The length of resin tags originated from self etching primers has been shown to contribute little to the bond strength of resin to enamel, and bonding is mainly attributable to the ability of resin in penetrating between the enamel crystallites and rods¹³.

The acidic primer promotes a selective dissolution of enamel prisms, creating micro-porosities for resin penetration¹⁶. Some studies have related that self-etching primers present bond strengths lower than or similar to those presented by one-bottle adhesive systems with acid etching¹⁶. This can be explained because the resin fails to penetrate deeply into enamel prisms¹⁶. As a consequence, the adhesion between enamel and resin fails after some time in function in the mouth¹⁶.

It was observed that self-etching primers did not create a deep etch pattern, differently from the phosphoric acid application only²⁰. Empty areas in the hybridized enamel region in the enamel/resin interface were also observed¹⁹. The balance between demineralization depth and extension of monomer penetration of the self-etching primers is the key to a good quality adhesion between enamel and resin²³.

Self-etching primers had limited demineralization and impregnation depths because of wet dentin and ionic effects of high calcium and phosphate concentrations, which limited the apatite crystals dissolution²⁸. Due to the high enamel mineral content, this effect should be considered when self-etching primers are applied in enamel.

Even though the self-etching primers do not have the same performance as that of conventional systems, they present high bond strength values and good clinical outcomes^{10,24}. Despite these opinions, the self-etching primers were not better than the conventional systems when applied to dentin¹⁷. Hence, the application of conventional adhesive systems to enamel and dentin is considered a safer alternative.

CONCLUSION

1. Considering the Scotchbond Multipurpose Plus multi-bottle adhesive system, the polymerization with halogen light resulted in higher

bond strength values than those obtained with polymerization with LED.

2. Considering the Clearfil SE Bond self-etching primer, there were no statistically significant differences between polymerization with halogen light or with LED.
3. There were no statistically significant differences between the Scotchbond Multipurpose Plus adhesive polymerized with LED and the Clearfil SE Bond adhesive polymerized with halogen light or with LED.
4. The multi-bottle adhesive system presented higher bond strength values than the self-etching primer.

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