

Shear Bond Strength of Self-etching and Total-etch Adhesive Systems to Er:YAG Laser-irradiated Primary Dentin

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ABSTRACT

Purpose: The purpose of this study was to assess in vitro the shear bond strength of self-etching and total-etch adhesive systems to Er:YAG laser-irradiated primary dentin.

Methods: Forty crowns of primary canines were embedded in acrylic resin and mechanically ground to expose a flat dentin surface. The specimens were randomly assigned to 2 groups (N=20), according to the adhesive system: (A) Single Bond (SB); and (B) Adper Prompt (AP). Each group was divided into 2 subgroups (N=10), depending on the surface treatment: (1) conventional bonding protocol, as recommended by the manufacturers; and (2) irradiation of the dentin site with a 2.94- μ m wavelength Er:YAG laser, with a 300-mJ pulse energy and a 2-Hz repetition rate followed by the bonding protocol. In both groups, a 3-mm diameter dentin bonding site was demarcated, the adhesive systems were applied, and resin composite cylinders were bonded. After 24 hours in distilled water, shear bond strength was tested at a crosshead speed of 0.5 mm/minute.

Results: Means (in MPa) were: group A1=14.14(\pm 1.7); group A2=8.41(\pm 1.04); group B1=6.88(\pm 1.12); and group B2=4.19(\pm 0.7). Data were submitted to statistical analysis using 2-way analysis of variance and t test at 5% significance level.

Conclusion: Irradiation of primary dentin with the Er:YAG laser decreased the bond strength of total-etch and self-etching adhesive systems.

(J Dent Child 2009;76:67-73)

Received September 18, 2007; Last Revision November 23, 2007; Revision Accepted November 25, 2007.

KEYWORDS: ADHESIVE SYSTEMS, ER:YAG LASER, PRIMARY DENTIN

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Over the last 30 years, dentistry has experienced a remarkable scientific advance regarding the improvement of restorative materials and techniques. The advent of acid etching and further introduction of adhesive restorative systems have revolutionized the dental practice, modifying the principles of cavity preparation and allowing a greater preservation of sound dental structure and a more esthetic treatment. Despite major advances in adhesive dentistry, bonding to dentin and the complete sealing of the exposed dentinal surfaces remains problematic because of the highly hydrated and complex nature of this tissue.^{1,2}

To achieve adhesion and bond strength between dental tissues and restorative materials, the smear layer formed during dental tissue preparation should be either removed or modified,^{3,4} which is achieved with the demineralization of dentin either by a separate acid etching step or by the use of a self-etching adhesive system.⁵ The adhesive systems have been developed to act on the tooth substrate prepared by conventional techniques. More recently, however, newer methods for cavity preparation have become widespread, such as laser irradiation.

The Er:YAG laser was first used in dentistry by Hibst and Keller in 1989⁶ and has proved to be a promising system. It presents a notorious ability to remove dental hard tissues, with minimal injury to the pulp and without causing severe thermal side effects, such as cracking, melting, or charring of the remaining tooth structure and/or surrounding tissues.⁶⁻⁸ Due to the great water content in its composition, dentin substrate is a target tissue with a strong interaction with the Er:YAG laser beam, which produces an irregular surface with no smear layer and open dentinal tubules.⁶⁻⁹ Because the morphological appearance of lased dentin strongly differs from that of dentin prepared with dental burs attached to high-speed handpieces,⁹⁻¹¹ there has been a major interest to investigate the interaction pattern between the currently available adhesive systems and the laser-irradiated dentin.

In pediatric dentistry, there is a great need to reduce the clinical chairtime without compromising the quality of the work. The goals of the so-called self-etching adhesive systems were to simplify the bonding procedure and reduce the adhesive protocol's technique sensitivity by combining the acid-etching step and the priming step in a single procedure.¹² The patients usually report feeling comfortable during a dental treatment with lasers because of the reduced noise and decrease in pain sensitivity, which sometimes eliminate the need for local anesthetics.¹³⁻¹⁵ These characteristics are especially important while treating children. Thus, in view of the remarkable and increasingly widespread use of adhesive dentistry in pediatric patients and the increasing approach of laser technology in dental practice, it seems relevant to assess the interaction pattern of the adhesive systems with lased primary dentin substrate, given its physiological dynamics, heterogeneous composition, and complex tubular structure. Studies should be done to avoid extrapolation of results obtained with permanent teeth to primary teeth because structural and morphological differences between primary and permanent substrates might interfere with the adhesion mechanism.^{16,17} Therefore, this study's purpose was to assess the shear bond strength of a self-etching and a total-etch adhesive system to Er:YAG laser-irradiated primary dentin.

METHODS

This study was approved by the Ethics in Research Committee of the School of Dentistry of Ribeirão Preto, University of São Paulo, Ribeirão Preto, São Paulo, Brazil, and written

informed consent was obtained from all parents or legal representatives for tooth donation.

Forty sound primary canines exfoliated and caries free of 11- to 12-year-old children within a 6-month period and stored in 0.4% sodium azide solution at 4°C were selected for this study. Prior to use, the teeth were washed in running water to eliminate storage solution residues and were carefully cleaned with water/pumice slurry in prophylaxis rubber cups at low speed. When necessary, roots were sectioned 2 mm below the cemento-enamel junction. Crowns were embedded in polyester resin using polyvinyl chloride rings (2.1 cm diameter and 1.1 cm height). After resin polymerization, the rings were discarded and the teeth's buccal surfaces were ground with water-cooled no. 180- to 400-grit silicon carbide paper (Buehler Ltd, Lake Bluff, Ill) on a polishing machine (Struers A/S, Copenhagen, Denmark) to remove the overlying enamel and expose a flat dentin surface. To warrant the complete removal of enamel, the ground surfaces were viewed under a X20 magnifier. Additional wet grinding with no. 600-grit SiC paper was done for 30 seconds to produce a standard smear layer. Rubber cup prophylaxis was performed with water/pumice slurry for 10 seconds followed by copious rinsing and gentle air drying. A dentin bonding site was demarcated by attaching a piece of insulating tape with a 3-mm-diameter central hole to each specimen surface. Bonding site delimitation had 2 aims: to define a fixed test surface area, and to warrant that the resin composite cylinders could be further adhered precisely to treated dentin surface, thus avoiding accidental adhesion to the surrounding enamel.

The specimens were randomly assigned to 2 groups (N=20), according to the adhesive system:

- A. Single Bond (SB; 3M/ESPE, St. Paul, Minn), an ethanol-and-water-based, total-etch single-bottle bonding agent; and
- B. Adper Prompt (AP; 3M/ESPE), a self-etching agent.

Each group was divided into 2 subgroups (N=10), depending on the surface treatment:

1. conventional bonding protocol, as recommended by the manufacturers; and
2. irradiation of the dentin site with a 2.94- μ m wavelength Er:YAG laser, with a 300-mJ pulse energy and a 2-Hz repetition rate followed by the bonding protocol.

The Er:YAG laser device used was the Kavo Key Laser 2 model (Kavo Dental GmbH & Co, Biberach, Germany). The laser beam was delivered on noncontact, defocused mode, with a fine water mist at 1.5 mL/minute rate. A number 2051 handpiece (Kavo Dental GmbH & Co, Biberach, Germany), attached to the flexible fiber delivery system, was used. The irradiation distance was standardized using a custom-made apparatus consisting of a holder that positioned the handpiece in such a way that the laser beam was delivered perpendicular to the specimen surface at a constant working distance of 12 mm from the target

site. It also used a semi-adjustable base, to which the specimen was fixed with wax. One previously trained operator handled the apparatus micrometer screws in such a way that the semiadjustable base with the specimen was alternately moved in right-to-left and forward-to-backwards directions, thereby allowing the laser beam to provide an accurate irradiation of the entire dentin site. The irradiation time was 20 seconds.

The tested adhesive systems were carefully applied with disposable tips (EMIC Ltda, São Jose dos Pinhais, Ribeirão Preto, São Paulo, Brazil) to avoid excess and pooling of adhesive

along the edges of the insulating tape that could compromise the distribution of tension during the shear test and alter of results. In group A, the dentin was etched with a 35% phosphoric acid gel (3M/ESPE) for 10 seconds, rinsed thoroughly, blotted with absorbent paper to remove excess water. The adhesive system was applied, slightly thinned with a mild oil-free air stream and light-cured for 20 seconds with a visible light curing-unit with a 450 mW/cm² output (XL 3000, 3M/ESPE), as measured with a curing radiometer (Demetron Research Corp, Danbury, Conn). For group B, equal drops of bonding agents A and B were

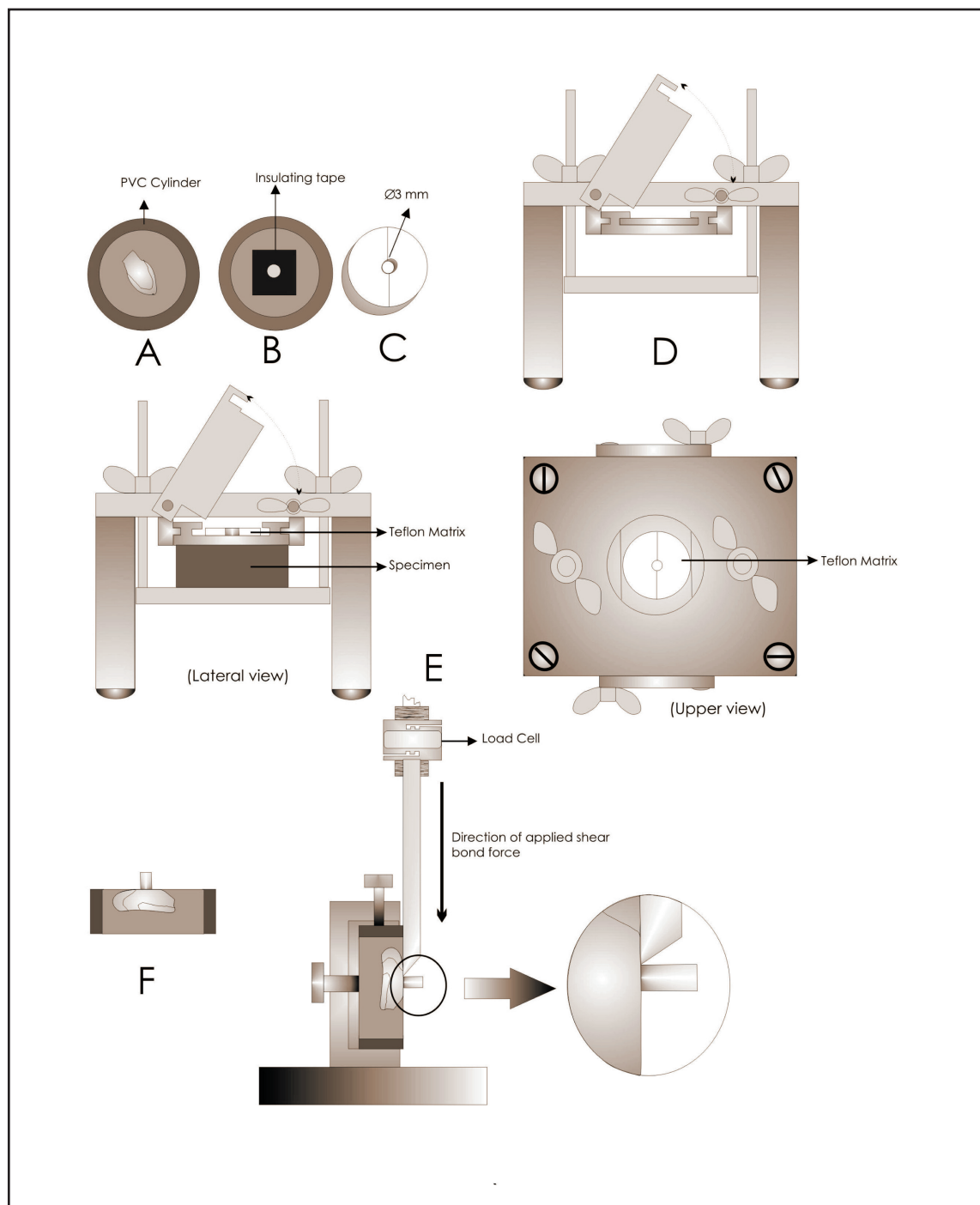


Figure 1a-f. Diagram of the study mechanism

dispensed into a mixing well and thoroughly mixed for 5 seconds until a homogeneous liquid mixture was obtained. The mixed, single-step adhesive was then applied to each dentin surface, rubbed in for 15 seconds, gently air air-dried to evaporated the volatile ingredients, and light-activated for 10 seconds.

After completing the bonding protocols, the specimens were individually fixed in a metallic clamping device (developed at Houston Biomaterials Research Center, University of Texas Dental Branch at Houston, Tex, and manufactured at the Precision Workshop of the School of Dentistry of Ribeirão Preto, University of São Paulo) that allowed keeping the test dentin surface parallel to a flat base. A split-bisected polytetrafluoroethylene jig was positioned on the tooth/resin block, thus providing a cylindrical cavity with 4 mm in height and 3 mm in diameter, which coincided with the demarcated dentin bonding site. Filtek Z250 hybrid light-cured composite resin (3M/ESPE) was inserted into the cavity in increments, each of which was polymerized for 40 seconds. As the cavity was completely filled, the specimen was removed from the clamping device and the jig was opened, leaving adhered to the dentin site a resin cylinder with same dimensions as that of the jig (4-mm high, 3 mm in diameter).

After 24-hour storage in distilled water at 37°C, the specimens were loaded in shear strength using a knife-edge blade in a universal testing machine (EMIC Ltda- Model MEM 2000, São José dos Pinhais, PR, Brazil) running at a crosshead speed of 0.5 mm/minute with 50 kgf load cell (Figure 1a-f). Bond strengths were recorded in kgf/cm and converted into MPa. Means and standard error were calculated, and data were analyzed by 2-way analysis of variance using a factorial design with adhesive system and surface treatment as independent factors. Multiple comparisons were done by *t* test at a .05 significance level. Fractured specimens were examined with a X40 stereomicroscope to assess the failure modes (adhesive, cohesive, or mixed). All examinations were done by a single examiner blinded to the groups to which the specimens belonged.

RESULTS

Bond strength data for nonlased and lased subgroups are shown on Table 1. Regarding the factor adhesive system alone, not considering the surface treatment performed, SB showed statistically higher bond strength means than AP ($P<.05$). Regarding the factor surface treatment alone, a significant ($P<.05$) decrease in bond strength was observed when dentin was irradiated with the Er:YAG laser before application of the total-etch adhesive system. No statistically significant difference ($P>.05$) was found between the bond strength recorded for the self-etching adhesive system on nonlased and lased primary dentin, although lased specimens showed numerically lower bond strengths.

Table 1. Shear Bond Strength (MPa) Means of the Control and Experimental Groups*

	Group A1 (SB)	Group A2 (SB + laser)	Group B1 (AP)	Group B2 (AP + laser)
Means±(SD)	14.14±1.7a	8.41±1.04b	6.88±1.12b	4.19±0.7bc

* Different letters indicate statistically significant difference and the same letter indicates statistical similarity among means at the 5% significant level.

Analysis of the bonding sites after the shear bond strength test revealed that an adhesive failure mode was predominantly observed in all groups and that cohesive failures occurred only in the nonlased Adper Prompt fractured specimens (10%).

DISCUSSION

The bond strength of adhesive systems is one of the major factors to be considered in the placement of esthetic restorations. An effective adhesion to tooth structure is of paramount importance to withstand the stresses resulting from polymerization shrinkage, thereby warranting retention and marginal integrity of restorations.¹⁸ Despite advances in the chemistry of adhesive systems, dentin remains a challenging substrate for bonding due to its heterogeneity.^{1,2}

Total etching with 30% to 40% phosphoric acid is one of the steps on the bonding protocol of several contemporary adhesive systems, and a 15-second acid-etching time is deemed ideal for conditioning of permanent tooth dentin.¹⁹ Permanent tooth dentin is more resistant to demineralization by phosphoric acid etching than primary tooth dentin.^{20,21} Studies with different types of adhesive systems have shown that bond strength to primary dentin is generally lower than that to permanent dentin.²²⁻²⁴ The chemical, physiological, and micromorphological differences between these substrates are thought to be responsible for the lower bond strength usually recorded in primary teeth.¹⁶ Primary dentin has lower hardness and mineral content than permanent dentin.¹⁷ The peritubular dentin of primary teeth is approximately 2 to 5 times thicker than that of permanent teeth.²⁵ Sumikawa et al¹⁶ reported that primary teeth have greater tubular numerical density and, hence, lesser availability of intertubular dentin, which may interfere with the establishment of a high-quality adhesion. Therefore, it has been suggested that shortening of the acid-etching time would yield the formation of a more functional hybrid layer in primary teeth.^{20,21}

Acid etching of dentin has always concerned clinicians and researchers. Factors inherent to dentin conditioning that might influence the bonding performance of adhesives in this substrate have been extensively discussed.²⁶ More recently, the concept of using the smear layer as a bonding substrate has been reintroduced. The rationale behind the self-etching adhesive systems is to minimize the adhesive

protocol's technique sensitivity by eliminating the acid-conditioning, rinsing, and drying steps. Fewer procedural steps mean less chairtime, which is particularly attractive in pediatric dentistry. In the present study, comparing the results on bond strength of the adhesive systems between those used in the conventional technique groups, when the smear layer is present, the adhesive system using a separate etching step showed greater bond strength. It is possible that the acidic primer evaluated in this study caused excessive demineralization of the dentin, as it has been shown that the lower the conditioner's pH, the deeper the dentin demineralization.²⁷ The resulting greater thickness of the hybrid layer and the subsequent lack of complete penetration of the adhesive resin into demineralized dentin may have contributed to the lower bond strength to primary dentin observed in this study. Silva-Telles et al²⁸ showed that composite resin (Filtek Z-250) restorations bonded with Prompt L-Pop to primary dentin presented interfacial gaps more frequently than those bonded with Single Bond.

The use of Er:YAG laser irradiation for dental applications has been increasingly widespread in the last few years. This technology has been presented as a viable option to replace the conventional high-speed air turbine and low-speed drills, offering improved patient comfort by reducing the pressure, heat, vibration, and noise associated with rotary dental instruments.^{13,29} These characteristics are of particular interest for pediatric dentistry.

The Er:YAG laser is an instrument for ablation of dental hard tissue due to its 2.94- μ m wavelength light emission, which coincides with the absorption peak of water and hydroxyapatite. It can remove enamel and dentin more effectively than other laser systems.^{6,7,30} The ablation of tooth structure is achieved via a thermomechanical interaction. Because the lased substrate is not completely vaporized, but only disintegrated into fragments, most incident radiation is consumed in the ablation process. This leaves very little residual energy for adverse thermal interactions with the pulp and surrounding soft and hard tissues.^{6,31} The higher water content and the relatively predominant organic composition of dentin potentializes the action by the Er:YAG laser. This is even more accentuated in primary tooth dentin³² due to the structural, chemical, and morphological characteristics of this substrate.^{16,17,25}

Several characteristics of the lased dentin have previously been considered as advantageous for resin bonding. They include the formation of a microscopically rough substrate surface without demineralization, open dentinal tubules without entrance enlargement, and no smear layer production.^{9,33,34} Laser ablation, however, has been reported to cause other surface alterations. Some authors have examined resin-dentin interfaces in permanent teeth and found microcracks below the hybrid layer, indicating that subsurface damage was caused by Er:YAG irradiation.^{35,36} The potential impact of the Er:YAG laser on the collagen network has not yet been clearly disclosed. It remains unclear whether the microstructural alteration and microrupture of collagen fibers caused by laser irradiation would

actually compromise the interaction of adhesive systems with laser-treated dentin, which would negatively affect the bond strength. Furthermore, it is also important to highlight the action of laser irradiation on the mineral components of dentin. Although SEM observations have revealed that Er:YAG laser-treated dentin shows little to no smear layer and open dentinal tubules, the laser beam does not effectively act on the peritubular dentin. Hence, it is not able to enlarge tubules' openings.^{9,34,37} This peculiar morphology may affect hybridization and negatively influence the acid-reactivity of lased dentin, as the etchant or acidic monomer may not be as efficient at dissolving the mineral components of the superficial laser-irradiated dentin, thus affecting the bonding effectiveness.

It is important to emphasize that all adhesive materials have been developed to be applied to tooth substrate prepared with rotary instruments and treated with conventional techniques. In spite of the well-known cutting efficacy of the Er:YAG laser, it has been demonstrated that laser irradiation does not eliminate the need for acid-etching. Resin materials bonded to nonetched lased surfaces lack the seal obtained with acid conditioning.^{36,38,39} Monghini et al⁴⁰ reported that dentin acid etching was beneficial for adhesion in an *in vitro* study that assessed the influence of the Er:YAG laser on shear bond strength of a total-etch adhesive system to lased primary dentin.

Therefore, in the present study, laser irradiation was followed by phosphoric acid etching, except for group B2, in which a self-etching system was used (Adper Prompt). The groups in which the lased dentin was etched with phosphoric acid had higher shear bond strength. It is likely that the acid-etching and water-rinsing steps appeared to have eliminated the surface laser-modified layer. Acid etching, however, is not able to eliminate the laser-modified layer completely.⁴¹ The thermomechanical effects produced by laser irradiation probably extend into the subsurface dentin and undermine the resin-dentin interface's integrity.⁴² This probably accounted for the lower shear bond strength compared to nonlased acid-etched dentin in the present study. Previous reports have explained the low bond strength obtained in Er:YAG laser-treated permanent dentin as a consequence of physiochemical changes caused by laser energy in the tissues.^{10,38} Schien et al¹¹ examined the interaction pattern formed between dentin and resin (Single Bond and Z100 - 3M Dental Products, St Paul, MN, USA) on cavities prepared with the Er:YAG laser. They reported that the dentin-resin interfacial aspect of acid-etched irradiated dentin showed thin tags and scarce hybridization zones.

The self-etching adhesive system approach allowed a considerable decrease in the operative time, which is of particular interest for restoration of teeth that cannot be adequately isolated, as commonly occurs in pediatric dentistry. Nevertheless, in spite of their expected good bonding ability and advertised benefits, some of the currently available self-etching primers do not perform as well as the total-etch adhesive systems in laboratorial studies.

This leads to the assumption that an underdetermined clinical outcome may be expected as well. Therefore, given the great applicability of adhesive dentistry in pediatric patients, future studies should be conducted to investigate the bond strength, characteristics of resin/dentin interface, and type of interaction between contemporary self-etching adhesive systems and primary dental substrates.

Focusing specifically on the Er:YAG laser, long-term studies have yet to determine how the morphological changes produced by irradiation might affect the performance of adhesive materials. It seems unlikely that the formation of an interdiffusion zone between the resin monomers and the irradiated intertubular dentin (ie, a hybrid layer) occurs with the same characteristics observed on conventionally prepared and treated dentin surfaces. Indeed, most conclusions drawn from studies on the mechanism of bonding to lased tooth surfaces (especially primary dentin) are still based on empiric observations and speculations. Further research is required to determine the best adhesive protocol for laser-prepared dentin and support the development of materials that are able to interact properly with this substrate. This will help establish the basis for rational assessment and applicability of laser technology in restorative pediatric dentistry.

The present in vitro study assessed the shear bond strength of self-etching and total-etch adhesive systems to Er:YAG laser-irradiated primary dentin. The lack of studies testing the same methodology, technology, and materials on primary teeth was a hindrance to stating a reliable comparison between this study's outcomes and the available data. Although the findings obtained from permanent teeth have been assumed to apply to primary teeth, the existence of remarkable differences between primary and permanent substrates must be considered.

CONCLUSIONS

Based on this study's results and within the limitations of an in vitro investigation, it may be concluded that the irradiation of primary dentin with an Er:YAG laser adversely affected the interaction pattern of total-etch and self-etching adhesive systems with the lased substrate and yielded a significant decrease in bond strength.

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