Influence of light-activated and auto- and dual-polymerizing adhesive systems on bond strength of indirect composite resin to dentin

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Statement of problem. Clinicians must be aware of the bonding effectiveness of auto- and dual- polymerizing adhesive systems before choosing the material and technique of cementing inlay/onlays to dentin. An inadequate choice may compromise the success of indirect restorations.

Purpose. This study compared the microtensile bond strength (MTBS) of indirect composite resin bonded to dentin by light-activated, autopolymerizing, and dual-polymerizing adhesive systems.

Material and methods. Occlusal dentin surfaces of 36 human third molars were exposed and flattened. Teeth were assigned to 1 of the following 6 groups (n=6) of adhesive luting systems: 2 dual-polymerizing systems (Scotchbond Multipurpose Plus/Rely X [SBMP] and Prime & Bond NT Dual Cure/Enforce [PBDC]); 1 autopolymerizing system (ED Primer/Panavia F [EDP]); and 3 light-activated systems (control groups) (Adper Single Bond/Rely X [SB], Prime & Bond NT/Enforce [PB], and Clearfil SE Bond/Panavia F [CF]). The restorative materials were applied according to manufacturer's directions. A 2-mm-thick prepolymerized composite resin (Clearfil APX) disc was cemented with the resin cements on the bonded dentin. Teeth were stored in water at 37°C for 24 hours. Afterwards, teeth were sectioned both mesial-distally and buccal-lingually to obtain multiple bonded beam specimens with 0.8 mm^2 of cross-sectional area. Each specimen was tested in tension at a crosshead speed of 0.5 mm/min until failure. Data (MPa) were analyzed by 1-way analysis of variance and the Tukey post hoc test (α=.05). Failure patterns of tested specimens were analyzed using scanning electron microscopy.

Results. Mean MTBS values (MPa) for experimental groups were as follows: SBMP, 32.89 ± 3.26^{a}; SB, 26.74 ± 7.45^{ab}; PB, 26.11 ± 4.48^{ab}; CF, 25.30 ± 6.42^{ab}; EDP, 16.82 ± 5.53^{bc}; PBDC, 11.20 ± 5.89^{c} ($P<.001$). Groups with similar lowercase letters were not significantly different. Failure pattern of fractured specimens varied according to the polymerization mode.

Conclusion. The autopolymerizing system and one of the dual-polymerizing systems were as effective as the light-activated systems in bonding indirect composite restorations to dentin. (J Prosthet Dent 2006;96:115-21.)

CLINICAL IMPLICATIONS

Light exposure is important for adhesive systems to promote higher bond strength in indirect composite resin restorations. The effectiveness of dual- and autopolymerizing bonding systems for indirect restorations may be more related to the material than the mode of polymerization.

Esthetic results and longevity of indirect composite resin restorations depend on each step of the clinical and laboratory procedures. Cementation is the most critical step and involves the application of both adhesive system and resin luting agent.[1,2] The clinical protocol for placing laboratory-processed composite resins includes the use of dual-polymerizing resin cements; however, the dentin bonding adhesive system can be light-activated, autopolymerizing, or dual-polymerizing.[3,4]

Some studies have suggested the application of adhesive systems that are capable of reaching complete polymerization by light exposure prior to placement of the indirect restoration.[5-9] However, tooth preparation for indirect restorations may result in areas that cannot be effectively reached by the light from the light-polymerizing unit. When an indirect porcelain or composite onlay is placed, the material opacity may inhibit sufficient light energy from being transmitted to the resin luting agent[10] and to the adhesive system. The incompletely polymerized areas either within the hybrid layer or at the adhesive layer may allow the diffusion of

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Table I. Experimental groups

Dentin surface treatment		Composite resin surface treatment	
Adhesive system (lot no.)	**Mode of polymerization**	**Silanization (lot no.)**	**Resin cement (lot no.)**
Adper Single Bond (3MJ)	Light-activated	Ceramic Primer (7KH)	Rely X (EEEG)
Scotchbond Multipurpose Plus (Primer: 3AJ) (Catalyst: 3AP/Activator: 7546)	Dual	Ceramic Primer (7KH)	Rely X (EEEG)
Prime & Bond NT (0406000421)	Light-activated	Silane Coupling Agent (Primer) 176675	Enforce (558)
Prime & Bond NT Dual Cure (Adhesive: 0406000421) (Activator: 121405)	Dual	Silane Coupling Agent 176675	Enforce (558)
Clearfil SE Bond (Bond: 00447) (Self-etching primer: 00434B)	Light-activated	Clearfil Porcelain Bond Activator + SE Primer 00593B/00434B	Panavia F 00021B/00229B
ED Primer (Primer A: 00204A) (Primer B: 00054E)	Auto	Clearfil Porcelain Bond Activator + SE Primer 00593B/00434B	Panavia F 00021B/00229B

water,[11,12] which may impair the bond strength at those areas and compromise the longevity of adhesive restorations.[13] Thus, dual-polymerizing or autopolymerizing adhesive systems are indicated for direct and indirect restorations to ensure proper polymerization of the adhesive resin in such areas.[14,15]

A high-quality hybrid layer requires optimal infiltration of adhesive monomer into enamel and dentin substrates as well as optimal polymerization within substrate.[16-18] Laboratory and clinical trial studies usually investigate the mechanical properties of light-activated bonding agents;[19-23] however, there is little published information about bonding of autopolymerizing and dual-polymerizing adhesive systems that confirm their effectiveness. Thus, the objective of this study was to evaluate the effects of light-activated, autopolymerizing, and dual-polymerizing adhesive systems and resin cement on bond strength of indirect composite resin to dentin. The null hypothesis tested was that bond strength is not influenced by dentin bonding systems with different polymerization modes.

MATERIAL AND METHODS

Thirty-six erupted human third molars that were stored in thymol-saturated water at 5°C for no more than 3 months were used in this study. The teeth were obtained after informed consent was provided by the patients and the protocol was approved by the Local Ethical Committee in Research of the Piracicaba School of Dentistry/UNICAMP, Brazil (114/2004). Teeth were transversally sectioned in the middle of the crown with a diamond blade (Isomet; Buehler Ltd, Lake Bluff, Ill) under water irrigation, exposing areas of middle coronal dentin. The exposed dentin surfaces were wet-polished with 600-grit SiC (3M do Brasil Ltd, Sumare, Sao Paulo, Brazil) paper for 10 seconds to create a flat surface with a standard smear layer[21,24] before being bonded with the adhesive systems.

Indirect bonding restorative procedures

Teeth were assigned to 1 of the following 6 groups (n=6) (Table I) of adhesive luting systems: 2 dual-polymerizing systems (Scotchbond Multipurpose Plus/Rely X [SBMP] and Prime & Bond NT Dual Cure/Enforce [PBDC]); 1 autopolymerizing system (ED Primer/Panavia F [EDP]); and 3 light-activated systems (control groups) (Adper Single Bond/Rely X [SB], Prime & Bond NT/Enforce [PB], and Clearfil SE Bond/Panavia F [CF]) (Table II). Adper Single Bond, Prime & Bond NT, and Clearfil SE Bond adhesive systems were applied to dentin and light-activated for 10 seconds (XL 3000; 3M ESPE) prior to indirect composite resin cementation, while no adhesive photoactivation was performed prior to cementation when Scotchbond Multipurpose Plus, Prime & Bond NT Dual Cure, and ED Primer were applied to the dentin surface.

Thirty-six prepolymerized resin composite discs (2 mm thick and 10 mm in diameter; shade A2) were prepared with composite resin (Clearfil APX; Kuraray Medical Inc) to simulate laboratory-processed composite resin restorations. One surface of each composite resin disc was abraded with 50-μm aluminum oxide particles for 15 seconds (Microetcher II; Danville Materials, San Ramon, Calif) and silanated with coupling agents[25] (Table I). The adhesive systems, silane, and resin cements were manipulated and applied according to manufacturers' instructions.

The mixed composite resin pastes were applied to the airborne-particle–abraded surface of the composite disc, which was positioned and fixed to the treated dentin surface under a load of 500 g for 5 minutes. The light-polymerizing unit tip was positioned against the composite disc, and each specimen was light activated for 40 seconds with a light-polymerizing unit (XL 3000; 3M ESPE) operated at 600 mw/cm^2. Afterwards, a 3-mm-thick block of autopolymerized composite resin (Concise; 3M do Brasil Ltd) was added to the untreated composite surface.

Table II. Composition of adhesive systems used in this study

Adhesive systems	Composition	Manufacturer
Adper Single Bond	Adhesive: water, ethanol, Bis-GMA, HEMA, UDMA, bisphenol A glycerolate, polyalkenoic acid copolymer, dimethacrylate. Echant: water; 35% phosphoric acid; synthetic amorphous silica.	3M ESPE, St. Paul, Minn
Clearfil SE Bond	SE-Primer: MDP, HEMA, CQ, N,N-diethanol p-toluidine, hydrophilic dimethacrylate and water (pH=2). SE-Bond: MDP, Bis-GMA, HEMA, hydrophobic dimethacrylate, CQ, N,N-diethanol p-toluidine and silanated colloidal silica.	Kuraray Medical Inc, Okayama, Japan
Prime & Bond NT Dual Cure	Adhesive: di- and trimethacrylate resins, PENTA, photoinitiators, stabilizers, nanofillers - amorphous silicone dioxide; cetylamine hydrofluoride, acetone (<70% wt). Autopolymerizing activator: acetone (<65% wt); ethyl alcohol (<45% wt); sodium p-toluenesulfinate. Etchant: water, 34% phosphoric acid, silicon dioxide, surfactants, blue colorant.	Dentsply Caulk, Milford, Del
Prime & Bond NT	Adhesive: di- and trimethacrylate resins, PENTA, photoinitiators, stabilizers, nanofillers - amorphous silicone dioxide; cetylamine hydrofluoride, acetone (<70% wt).	Dentsply Caulk
Scotchbond Multipurpose Plus	Activator: ethyl alcohol; sodium benzenesulfinate. Primer: water; HEMA; copolymer of acrylic and itaconic acids. Catalyst: bisphenol A diglycidyl ether dimethacrylate; HEMA; benzoyl peroxide. Etchant: water; 35% phosphoric acid; synthetic amorphous silica.	3M ESPE
ED Primer	Primer A: 10-methacryloyloxydecyl dihydrogen phosphate; hydroxyethyl methacrylate, ethylene glycol methacrylate; N, N-di-(2-hydroxy-ethyl)-p-toluidine; N-methacryloyl 5-aminosalicylic acid; water. Primer B: benzenesulfinic acid, sodium salt; N, N-di-(2-hydroxyethyl)-p-toluidine; N-methacryloyl 5-aminosalicylic acid; water	Kuraray Medical Inc

Bis-GMA, Bisphenol-A glycidyldimethacrylate; *HEMA*, 2-hydroxyethyl methacrylate; *UDMA*, urethane dimethacrylate; *MDP*, 10-methacryloyloxydecyl dihydrogen phosphate; *CQ*, camphorquinone; *PENTA*, dipentaerythritol penta acrylate monophosphate.

Microtensile bond strength test

The restored teeth were stored in distilled deionized water at 37°C for 24 hours and were vertically, serially, sectioned into several 0.7-mm-thick specimens with a diamond blade (Isomet; Buehler Ltd). Each specimen was further sectioned to produce a specimen with a cross-sectional area of approximately 0.8 mm^2. Four beams were selected from each restored tooth. Each beam was fixed to the grips of a testing device with cyanoacrylate (Zapit; Dental Venture of America, Corona, Calif) and tested in tension in a universal testing machine (4411; Instron Co, Canton, Mass) at 0.5 mm/min until failure. Afterward, the specimens were carefully removed from the testing machine 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 (Starrett 727-6/150; Starrett, Sao Paulo, Brazil) to calculate microtensile bond strength (MTBS), which was expressed in MPa. Mean values of the 4 beams were calculated for each restored tooth.

This study aimed to evaluate only the effectiveness of all materials following the manufacturers' instructions. Therefore, as the mode of polymerization was not an alternative treatment, a 1-way analysis of variance (ANOVA) and Tukey post hoc test were performed to compare the MTBS of all adhesive systems (α=.05).

Table III. One-way ANOVA

Source	*df*	Type I sum of squares	Mean square	F	*P*
Product	5	1823.99	364.79	11.36	<.001

df, Degrees of freedom.

Failure modes

Fractured surfaces of tested specimens were allowed to dry at 37°C in an oven (MA-032/1; Marconi Equipment Ltd, Piracicaba, SP, Brazil) overnight. The surfaces were sputter-coated with gold (MED 010; Balzers AG, Balzer, Liechtenstein) and observed under a scanning electron microscope (VP- 435; LEO, Cambridge, UK) to analyze the failure pattern. The failure patterns were classified as cohesive in adhesive or dentin, or cohesive along the bonding resin/hybrid layer interface. The failure pattern was also classified as mixed when adhesive resin or resin cement and hybridized dentin could be observed on the fractured specimen.

RESULTS

One-way ANOVA indicated that there were significant differences among groups ($P<.001$) (Table III). Mean values of MTBS for experimental groups are displayed in Table IV. The Tukey post hoc test showed that the dual-polymerizing adhesive system, Scotchbond Multipurpose Plus (SBMP), exhibited higher bond strength than the other dual-polymerizing (Prime & Bond NT Dual Cure [PBDC]) and autopolymerizing (ED Primer [EDP]) systems tested ($P<.001$). However, no significant differences were observed among SBMP and the light-activated Adper Single Bond (SB), Prime & Bond NT (PB), and Clearfil SE Bond (CF) systems. No significant differences were found among EDP, SB, PB, and CF. However, PBDC demonstrated significantly lower MTBS than the light-activated adhesive systems and SBMP ($P<.001$). No significant differences in MTBS were observed between PBDC and EDP.

Mixed failure in hybridized dentin, adhesive layer, and resin cement was the most predominant failure pattern observed for SBMP and all light-activated adhesive systems (Fig. 1). The failure patterns observed for the dual-polymerizing PB and the autopolymerizing EDP groups were cohesive along the bonding resin/hybrid layer interface, with failures occurring predominantly within and at the bottom of the hybrid layer (Fig. 2).

DISCUSSION

The polymerization of adhesive resin initiated by light exposure before cementation may ensure better mechanical properties for the polymer network inside the dentin, improving bond strength. As photoactivation can produce a thick adhesive layer that may compromise the adaptation of indirect restorations, autopolymerizing and dual-polymerizing adhesive systems are indicated to enhance marginal fit.[8,14] In the present study, significant differences in MTBS were found among the experimental groups. The results demonstrated that the bond strength of the dual-polymerizing SBMP was significantly different from that exhibited by the other dual-polymerizing and autopolymerizing adhesive systems. Furthermore, the MTBS of chemically polymerized SBMP was similar to the light-activated adhesive systems, which in turn exhibited higher MTBS than PBDC and were similar to the autopolymerizing EDP mean values. Thus, the null hypothesis that bond strength is not influenced by dentin bonding systems with different polymerization modes was rejected.

Table IV. MTBS values (mean ± SD) for experimental groups (MPa)

Adhesive luting system	MTBS	Tukey test
Scotchbond Multipurpose Plus/Rely X	32.9 ± 3.3	A
Adper Single Bond/Rely X	26.7 ± 7.5	AB
Prime & Bond NT/Enforce	26.1 ± 4.5	AB
Clearfil SE Bond/Panavia F	25.3 ± 6.4	AB
ED Primer/Panavia F	16.8 ± 5.5	BC
Prime & Bond NT Dual Cure/Enforce	11.2 ± 5.9	C

Mean values followed by different uppercase letters are significantly different at $P<.05$.

The evaluated light-activated bonding systems (SB, PB, and CF) are primarily indicated for direct restorations, although they can be used in indirect procedures. Some authors concluded that photoinitiated polymerization of the adhesive systems is necessary to ensure optimum bonding to dentin.[7-9] This was confirmed in this study based on the fact that the bond strengths of photoinitiated adhesive systems were not different from the highest mean bond strength obtained when the dual-polymerizing adhesive system, SBMP, was used. It may also be speculated that the 2-mm-thick indirect composite resins cemented over bonded dentin allowed adequate light intensity to reach the composite resin and the adhesive resins, increasing the degree of conversion and, consequently, the bond strength.

The 2 dual-polymerizing bonding systems evaluated in this study exhibited opposite results. The SBMP adhesive system showed high mean MTBS, while PBDC showed the lowest. The SBMP has primer and bonding agents in separate bottles. After rinsing and drying, the residual water content from dentin is reduced following primer application. Consequently, when the

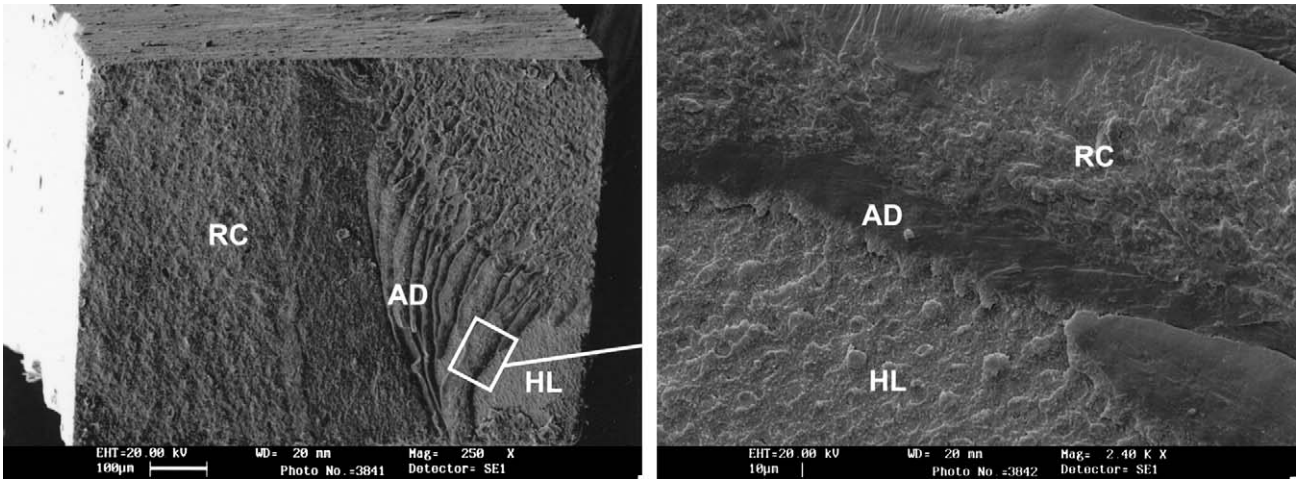

Fig. 1. Left, Representative SEM image (original magnification ×250) of failure pattern observed for SBMP and light-activated adhesive systems. Fracture occurred predominantly within adhesive layer *(AD)* and at bottom of resin cement layer *(RC)*. **Right,** Only small areas of fracture within hybrid layer were seen *(HL)*.

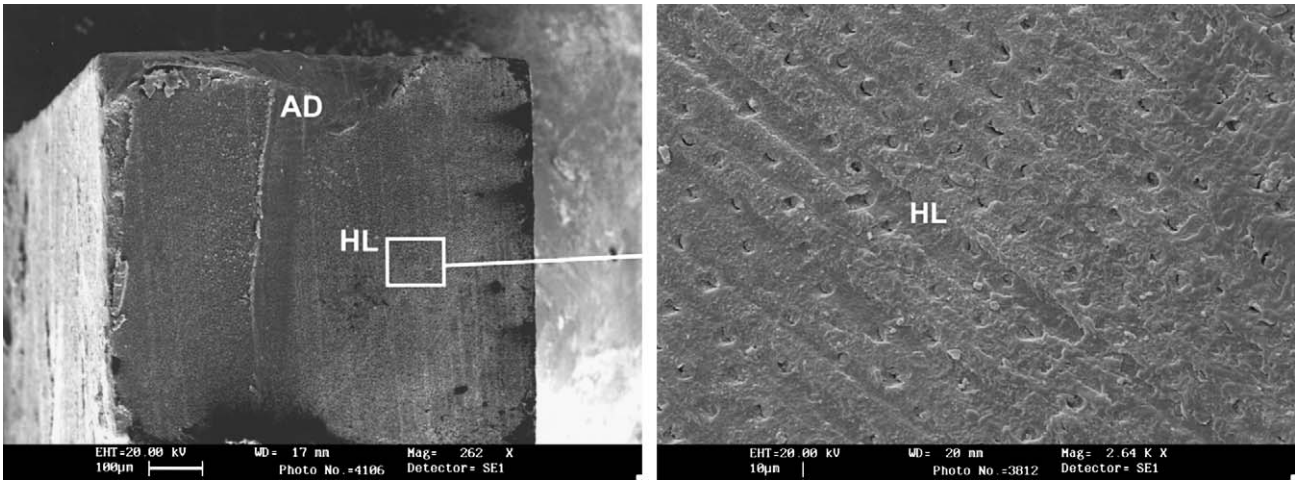

Fig. 2. Left, Representative SEM image (original magnification ×262) of failure pattern observed for EDP and PBDC. **Right,** Most predominant failure mode occurred at bottom of hybrid layer *(HL),* although some areas corresponding to fracture within adhesive layer *(AD)* were also found.

hydrophobic adhesive resin is applied to dentin, the contact between water and the adhesive resin is minimal, avoiding the monomer phase separation and the dissolution of components. Thus, these adhesive systems may be capable of reaching a high degree of conversion and, consequently, high bond strength when they are applied to dentin.[18]

However, PBDC consists of a mixture between 1-bottle PB and an activator solution. According to the manufacturer's instructions, the adhesive system must be photoactivated before the placement of the resin cement. However, in this study, PBDC was left in the unpolymerized state before the resin cement application. The reason for this procedure was to evaluate the effectiveness of the dual-activated adhesive system when photoactivation is compromised or is not performed. The low MTBS observed may be evidence of the limited efficiency of the activator component in allowing the polymerization of the adhesive/resin cement interface, as well as the adhesive resin within the hybrid layer.

Another reason for the low MTBS observed when PBDC was used may be related to the composition of the bonding agent and activator components. As both bonding agent and activator solution have high content of organic solvents, such as acetone and ethyl alcohol (Table II), the mixture of the bonding agent and activator may create a solution with a low content of monomers/activator components and high concentration of organic solvents. Cho and Dickens[19] reported that the increase in the initial acetone content of single-solution

dentin bonding agents resulted in thinner adhesive layers and lower dentin tensile bond strengths. The authors also indicated that higher acetone concentration in such bonding solutions may, in spite of the drying step, allow the residual solvent in the adhesive resin to remain. The residual content of organic solvent may have promoted the phase separation between hydrophobic and hydrophilic components and reduced free-radical polymerization initiated by light exposure, as well as aromatic sodium sulfinate, the autopolymerizing initiator. Thus, resin polymerization of PBDC may have been compromised and the mechanical properties of the adhesive resins impaired as a consequence. Other studies have also described similar results for PBDC.[21,22]

The EDP exhibited similar MTBS to the 3 light-activated adhesive systems evaluated in this study. The EDP contains only an autopolymerizing catalyst system (chemical initiator) and combines etching, priming, and bonding into a single procedure. When Panavia F resin cement is applied to the EDP-treated dentin surface, polymerization via an autopolymerizing mechanism occurs because aromatic sulfinate salts from EDP diffuse into the polymerizing resin cement. Such chemical interaction allows the polymerization reaction to occur even without light exposure.[2] However, the hybrid layer formed by EDP has a higher concentration of hydrophilic and ionic resin monomers, which increases water diffusion into the adhesive resin.[10,11] This phenomenon can be confirmed by transmission electron microscopy (TEM) analysis, which usually shows blisters related to either the presence of water permeated from the dentinal tubules or incompletely polymerized regions as a result of the entrapment of water within the primer layer.[12] Moreover, the slow polymerizing rate of the autopolymerizing adhesive resin may have allowed the increase in water diffusion within the adhesive layer and, consequently, compromised the polymerization reaction as well as the EDP mechanical properties. These hypotheses may explain why the MTBS values of the EDP adhesive system were as low as those exhibited by the PBDC group and, also, why they were lower than other autopriming or etch-and-rinse adhesive systems demonstrated in other studies.[20,21]

The differences in the failure pattern may confirm the hypothesis that the polymerization reaction of the adhesive resin within the hybrid layer and at the adhesive layer was compromised when either EDP or PBDC was used. The failures in specimens restored with these adhesive systems occurred predominantly within the hybrid layer (Fig. 2), which in turn may be considered the weakest portion of the bonded interface. Although many other factors may affect the mechanical properties of the hybrid layer, it is possible that such differences in the failure patterns may be indirect evidence of the differences in the degree of conversion of the adhesive resins infiltrated into the dentin. However, specimens restored with SBMP and the light-activated adhesive systems showed a mixed failure pattern, which was characterized by the presence of resin cement, adhesive layer, and the top of the hybrid layer on the fractured surface (Fig. 1). Further studies are needed to evaluate the potential of monomer conversion of these adhesive systems and resin cements when they are applied to dentin.

This in vitro study evaluated the MTBS of dual-polymerizing resin cements and adhesive systems in laboratory conditions. Therefore, factors such as dentinal fluid movement and internal stress related to the cavity configuration for indirect restorations were not simulated with this methodology. Further studies are necessary to evaluate the influence of those factors on the mechanical properties of the adhesive interface created by such cementing systems bonded to teeth. According to the results of this study, when dual-polymerizing resin cements are used, the selection of the dental adhesive is important to avoid the early bonding failure. Thus, in some clinical situations, such as cementation of prefabricated posts and cementation of thicker composite or ceramic restorations and metal crowns,[10] the dual-polymerizing adhesive resins must rely on the activation provided by autopolymerizing reactions to ensure the bonding durability of indirect bonded restorations.

CONCLUSION

Within the limitations of this study, the MTBS of the tested adhesive systems to dentin was material-dependent and indicated that 1 dual-polymerizing and all autopolymerizing systems tested demonstrated similar bond strength when using light activation only. The effectiveness of autopolymerizing and dual-polymerizing adhesive systems may be more closely related to product selection than to the polymerization mode used.

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Noteworthy Abstracts of the Current Literature

Clinical effect of different shade guide systems on the tooth shades of ceramic-veneered restorations
Hassel AJ, Koke U, Schmitter M, Beck J, Rammelsberg P. *Int J Prosthodont* 2005;18:422-6.

Purpose: The objective of this study was to investigate whether a systematically arranged shade guide system (Vita 3D-Master) allows clinicians to achieve a better shade match of a restoration, as compared to a conventional shade guide with a design based on empirical values (Vita Classical).
Materials and Methods: Fifty-nine restorations in 42 patients being treated by student clinicians were assessed. Using 1 of the 2 shade systems assigned randomly, each student independently determined the tooth shade. With the aid of a visual rating scale, the accuracy of the shade match of the finished restoration was assessed.
Results: All restorations whose shades had been determined with the 3D-Master could be placed without any further shade corrections. In contrast, almost 17% of restorations determined with the conventional system required subsequent shade modifications. The match of the shades selected with the 3D-Master was judged significantly better by the clinicians.
Conclusion: Within the limitations of the study, clinicians with less clinical experience who use a system that guides them through the shade-taking procedure in a relatively systematic manner will be more successful in selecting the correct tooth shade and in avoiding shade corrections. Clinical assessment of the restoration shades showed significant differences between the shade guide with a systematic design and that based on empirical values.—*Reprinted with permission of Quintessence Publishing.*