Dental Materials Journal 2008; 27(3): 376−385

Original Paper

Influence of Polymerization Mode on Degree of Conversion and Micropush-out Bond Strength of Resin Core Systems Using Different Adhesive Systems

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The aim of this study was to evaluate the influence of different polymerization modes on degree of conversion and micropush-out bond strength of dual-polymerized resin core systems to dentin by using two different adhesive systems. Two resin core systems, Rebilda DC and Build-It FR, were used in combination with total-etch (Adper Scotchbond Multi-Purpose) and self-etch (Clearfil SE Bond) adhesive systems. After treatment of dentin surfaces, resin core systems were applied into the cavities and subjected to different polymerization modes as follows: (1) chemical polymerization; (2) dual polymerization with standard mode of LED (Elipar FreeLight 2 LED); or (3) dual polymerization with exponential mode of LED. The cavities (n=10 per group) were tested in a universal testing machine. Degree of monomer conversion (DC%) was determined by Fourier transform infrared spectroscopy (n=5 cavities per group). ANOVA revealed that resin core ($p=0.002$), adhesive system ($p<0.001$), and polymerization mode ($p<0.001$) had significant effects on bond strength values. The degree of conversion for resin cores decreased significantly ($p<0.001$) when only chemical polymerization was employed.

Key words: Polymerization mode, Resin core, Adhesive system

Received Jul 20, 2007: Accepted Nov 13, 2007

INTRODUCTION

Core build-up materials are frequently used to repair endodontically treated teeth after excessive loss of the coronal portion and to stabilize the weakened parts of tooth structure[1]. In the face of a conspicuous horizontal loss of the clinical crown, such that only a ferrule of minimal thickness could be created in the remaining tooth structure, a post-and-core build-up is needed[2]. The most commonly used core materials are glass ionomers, resin composites, amalgam, and cast metal alloys[3]. Amongst which, resin composites are superior to glass ionomers and amalgam in that they enhance the retention and fracture resistance of posts. Furthermore, they have been shown to be more resistant to fatigue loading than cast metal posts and cores[4].

The use of resin-based materials with the appropriate adhesive system enhances retention and reinforcement characteristics that are essential to and beneficial in the treatment of weakened teeth[5]. To establish a strong and durable bond, which is necessary for the biomechanical aspect of the tooth-restoration system, appropriate treatment of the respective surfaces is crucial[6]. Current adhesive systems interact with the tooth structure using two different strategies: either remove the smear layer (total-etch technique) or maintain it as the substrate for bonding (self-etch technique)[7]. Self-etch adhesives do not require a separate acid-etch step and are based on the use of non-rinse acidic monomers that simultaneously condition and prime dentin[8,9]. Their bonding mechanism is based mainly on hybridization, which changes the chemical composition of the substrate surface layer of dentin; the exposed collagen layer is then filled with resin[10]. Moreover, self-etch adhesives reduce technique sensitivity by reducing the number of bonding steps, thereby also leading to shortened application time[9]. However, the efficiency of these simplified bonding systems has been more controversial than the total-etch system[11,12]. In particular, these simplified self-etch adhesives seemed to result in reduced bonding efficacy[13].

Recently, dual-polymerized resin composites have been introduced as both core build-up and luting materials[14]. Although the properties of modern resin composites have been improved, polymerization shrinkage still remains a clinically significant problem[15]. Shrinkage of resin composites occurs during polymerization and results in the failure of restorations, causing problems such as tooth fracture, marginal opening, microleakage, and secondary caries[16]. To reduce stress during polymerization, recent developments have focused on light curing protocols, such as soft-start, step-curing, or oscillating irradiation[17]. These curing modes are considered to enhance the degree of conversion and reduce internal stresses to preserve marginal integrity[18].

The degree of conversion (DC) is expressed as

a percentage of unreacted C=C bonds. It affects many properties of the resin composites, including mechanical properties, solubility, dimensional stability, color change, and biocompatibility[17]. To evaluate the DC of C=C bonds, spectroscopy methods have been used to determine the DC values because specific vibration bands can be used as internal standards[19].

A common and frequently used method to study the compatibility between an adhesive system and a resin composite is to evaluate the dentin bond strength[20]. Apart from bond strength, degree of conversion also provides additional information about polymerization. The purpose of this study, therefore, was to investigate the influence of polymerization modes and adhesive systems on micropush-out (μ-PO) bond strength of resin core systems to dentin. This study also determined the DC values of two resin core systems when cured using chemical polymerization *versus* polymerization using a light-emitting diode (LED) light curing unit in standard and exponential modes.

MATERIALS AND METHODS

Specimen preparation

Thirty intact, extracted, noncarious human molars, stored in 0.5% chloramin-T solution at 48°C for up to one month were used in this study. The teeth were debrided with a scaler (H6/H7 Scaler, Hu-Friedy, Chicago, IL, USA) and with pumice (Americos Industries Inc., Gujarat, India) using rotary brushes (Merssage Brush, Shofu Inc, Kyoto, Japan). Each tooth was ground occlusally with silicone carbide abrasive papers up to No. 1000 (Federation of European Producers of Abrasives (FEPA), Paris, France) under water cooling with a grinding machine (Struers RotoPol 11, Struers A/S, Rodovre, Denmark) to obtain a flat dentin surface. Thirty disks of 1.5 mm thickness were cut perpendicular to the tooth axis using a slow-speed diamond wafering blade (Ernst Leitz GmbH, Wetzlar, Germany). Five standardized occlusal cavities of 1.3 mm occlusal diameter, 1.2 mm cervical diameter were then prepared in each disk using a diamond bur (#801-016, Diatech Dental AG, Heerbrugg, Switzerland) in a high-speed handpiece under air-water spray coolant (Fig. 1).

Materials used in this study are listed in Table 1. One hundred and twenty cavities were divided into two adhesive groups: total-etching system (Adper Scotchbond Multi-Purpose Plus, 3M ESPE, St. Paul, MN, USA) *versus* self-etching system (Clearfil SE Bond, Kuraray Co. Ltd., Osaka, Japan) (n=60). Each adhesive group was further subdivided into two groups according to the resin core system: Build-It FR (Pentron Clinical Technologies, LLC, USA) *versus* Rebilda DC (VOCO, Cuxhaven, Germany) (n=30). Each of these groups was further divided into three more groups according to polymerization mode: chemical polymerization *versus* dual polymerization in standard or exponential mode of LED (Elipar FreeLight 2 LED, 3M ESPE, St. Paul, MN, USA) (n=10).

Bonding procedures were performed according to manufacturers' instructions (Table 1). Resin core

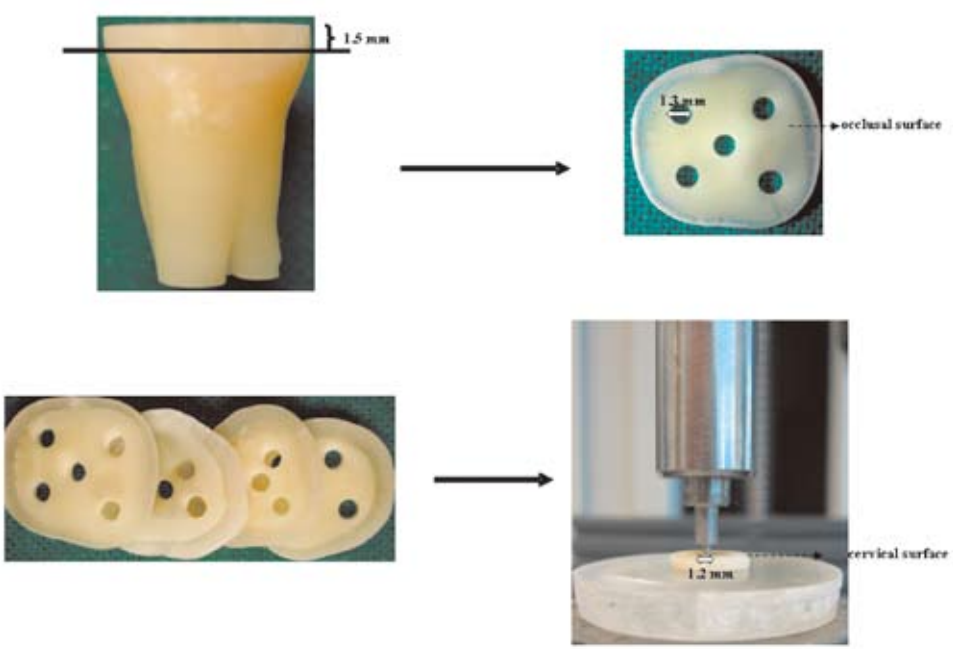


Fig. 1 Schematic illustration of specimen preparation and micropush-out test.

materials were applied and then either polymerized chemically or dual-polymerized with LED curing device in standard mode or exponential mode for 20 seconds from the occlusal surface prior to testing. The manufacturer stated that the standard mode provided full light intensity for the entire exposure period, whereas light in exponential mode increased to full light intensity over the course of five seconds. Specimens were stored in distilled water at 37°C for 24 hours.

Micropush-out bond strength

After measuring the thickness of each disk with a digital caliper (Liaoning MEC Group Co. Ltd., Mainland, China) for the push-out test, the specimens were mounted in a universal testing machine (Lloyd LRX, Lloyd Instruments Ltd., Fareham Hants, UK) and loaded at a crosshead speed of 1.0 mm/minute. Push-out force was applied from the bottom of the disk with a diameter of 1.2 mm because of convergence of the cavity. The force was recorded using a software (Lloyd Nexygen, Lloyd Instruments Ltd.), and the value used to calculate μ-PO strength. To express the bond strength data in MPa, the following formula was used[21]:

$$\mu\text{-PO bond strength(Mpa)} = \frac{\text{Maximum load (N)}}{\text{Adhesion area resin composite filling(mm}^2\text{)}}$$

Adhesion area of resin composite filling was defined as $(\pi r_1 + \pi r_2) \times L$. L could be calculated using the formula, $\sqrt{(r_1 - r_2)^2 + h^2}$, where r_2 is the occlusal radius, r_1 is the cervical radius, and h is the height of dentin disk.

Failure mode analysis

Failure modes were analyzed visually using a stereomicroscope (Wild M3B, Heerbrugg, Switzerland) and classified as follows: adhesive failure between resin core composite and dentin, cohesive failure within dentin, or cohesive failure within resin core composite and mixed type of failure.

SEM investigation of the specimens

One specimen was randomly selected from each group and prepared for SEM analysis. The debonded specimens from each group were gold sputter-coated (Bal-Tec SCD 050 Sputter Coater, Bal-Tec AG, Liechtenstein) and observed with a scanning electron microscope (JSM-5500, Jeol Ltd., Tokyo, Japan).

Table 1 Materials used in this study

Trade name	Type		Batch number	Composition	Bonding procedure
Adper Scotchbond Multi-Purpose	Total-etch three-bottle system	Etchant	6HK	37% phosphoric acid	Etch for 15s, rinse, dry gently
		Primer	6BG	HEMA[a], polyalkenoic copolymer, water	Apply primer dry gently for 5s
		Adhesive	6PP	Bis-GMA[b], HEMA[a]	Apply adhesive, light cure for 10s
Clearfil SE Bond	Self-etch two-bottle system	Primer	00648A	Water, MDP[c], HEMA[a], camphorquinone, hydrophilic dimethacrylate	Apply primer for 20s, mild air stream, apply bond, light cure for 10s.
		Adhesive	00920A	MDP[c], Bis-GMA[b], HEMA[a], N,N-diethanol-p-toluidine, hydrophobic dimethacrylate, silanated colloidal silica.	
Build-It FR	Fiber-reinforced, dual-cure core build-up material		143239	Bis-GMA[b], UDMA[d], HDDMA[e], silane treated bariumborosilicate glass fillers, chopped glass fibers, pigments with initiators, stabilizers and UV absorber	Place the auto-mix syringe tip at the bottom of the cavity, dispense the material into cavity
Rebilda DC	Dual-curing, flowable adhesive core build-up system		700115	Bis-GMA[b], UDMA[d], TEGDMA[f], BHT[g], BPO[h], silica, bariumborosilicate glass ceramic	Apply the material directly from the cartridge/Quickmix syringe

[a]HEMA, hydroxyethyl methacrylate; [b]Bis-GMA, bisphenol A-glycidyl dimethacrylate; [c]MDP, 10-methacryloyloxydecyl dihydrogen phosphate; [d]UDMA, urethane dimethacrylate; [e]HDDMA, 1,6-Hexanediol dimethacrylate, [f]TEGDMA, triethylenglycoldimethacrylate; [g]BHT, butylated hydroxytoluene; [h]BPO, benzoil peroxide

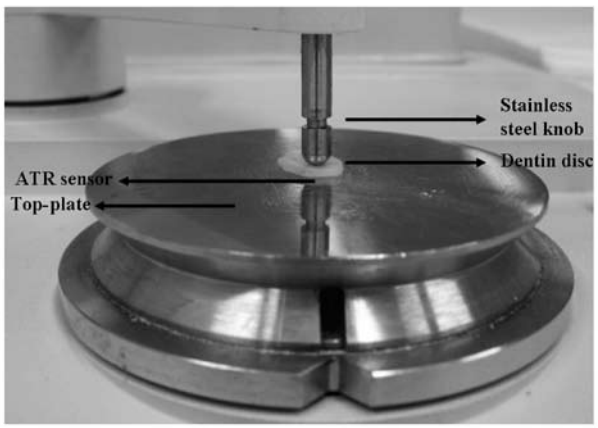


Fig. 2 Experimental set-up to evaluate degree of conversion.

Furthermore, one specimen from each adhesive system was stored in 2 M hydrochloric acid (HCl) for 48 hours to demineralize the tooth structure and reveal the resin tag system[22]. After extensive rinsing, the specimens were freeze-dried[23] and then gold sputter-coated for observation with a SEM (JSM-5500).

Degree of conversion analysis

The remaining 30 cavities were used for DC test. The experimental set-up of DC is given in Fig. 2. Resin core systems (Build-It FR and Rebilda DC) were introduced into the cavities and placed on the surface of a ZnSe ATR crystal (n=15 per group). The upper surface of the specimen was covered with a Mylar sheet and slightly pressed against the ATR sensor to ensure good contact with the specimen. Next, the resin core materials were either chemically polymerized or dual-polymerized in standard or exponential mode of LED for 20 seconds from the occlusal surface (n=5 per group). The light source was placed in contact with the surface.

Degree of monomer conversion (DC%) was monitored by Fourier transform infrared spectroscopy (FT-IR) (Spectrum One, Perkin Elmer, Beaconsfield Bucks, UK). First, the spectrum of the unpolymerized resin was measured, and then the infrared spectra were recorded at every 5.2 seconds for 15 minutes beginning after the mixing of the resin and using a reaction kinetics software (TimeBase V2, Perkin Elmer). During spectra recording, the resin was continuously in contact with the sensor. Thus, no oxygen was present on the resin surface to cause oxygen inhibition of the resin.

DC% was calculated from the aliphatic C=C peak at 1638 cm^{-1} and normalized against the aromatic C=C peak at 1608 cm^{-1} according to the following formula[24]:

$$DC\% = \left[1 - \frac{C_{aliphatic}/C_{aromatic}}{U_{aliphatic}/U_{aromatic}}\right] 100\%$$

where $C_{aliphatic}$=absorption peak at 1638 cm^{-1} of the polymerized specimen, $C_{aromatic}$=absorption peak at 1608 cm^{-1} of the polymerized specimen, $U_{aliphatic}$= absorption peak at 1638 cm^{-1} of the unpolymerized specimen, and $U_{aromatic}$=absorption peak at 1608 cm^{-1} of the unpolymerized specimen.

The fraction of remaining double bonds for each spectrum was determined by standard baseline techniques using a comparison of the maximum heights of aliphatic and reference peaks for the calculations.

Statistical evaluation

μ-PO bond strength data were statistically analyzed using three-way ANOVA for the following factors — resin core system, polymerization mode, and adhesive system. For the analysis of interaction effects among the factors, two-way ANOVA and Tukey's *post hoc* analysis were performed at a significance level of $p<0.05$. As for degree of conversion data, they were analyzed using two-way analysis of variance (ANOVA). All statistical analyses were performed using a statistical software package, SPSS (SPSS Inc., Chicago, IL, USA).

RESULTS

Table 2 presents the mean μ-PO bond strength values and standard deviations of the test groups in this study. Three-way ANOVA showed that polymerization mode ($p<0.001$), adhesive system ($p<0.001$), and resin core ($p=0.002$) had significant effects on μ-PO bond strength. Some interaction also existed between resin core system and polymerization mode ($p=0.038$). A statistically significant ranking for bond strength values was obtained as follows: chemical polymerization < standard mode = exponential mode ($p<0.05$). In all groups, bond strength value decreased with the use of self-etch adhesive system. The highest mean μ-PO value was seen in Group TRS (27.0±4.7 MPa), while the lowest was found in Group SBC (12.8±3.6 MPa).

Figure 3 shows the mean DC% values and the standard deviations of the luting resins at 15 minutes after the start of polymerization. Two-way ANOVA revealed significant differences between resin cores for the same polymerization mode ($p<0.001$). The interaction between factors was also significant ($p<0.001$). Additionally, the DC% of Build-It FR (62.9±0.8%) and Rebilda DC (68.5±1.3%) when polymerized chemically were significantly lower than the DC% values of these materials when polymerized

Table 2 Mean micropush-out bond strength values and standard deviations of the test groups

Group name	Group code	N	Mean	SD
Total-etching, Build-It FR, chemical polymerization	TBC	10	19.1[a,b]	3.2
Self-etching, Build-It FR, chemical polymerization	SBC	10	12.8[a]	3.6
Total-etching, Rebilda DC, chemical polymerization	TRC	10	22.3[b,c,d]	3.8
Self-etching, Rebilda DC, chemical polymerization	SRC	10	20.4[b,c]	3.2
Total-etching, Build-It FR, standard mode	TBS	10	26.4[c,d]	3.7
Self-etching, Build-It FR, standard mode	SBS	10	21.4[b,c,d]	4.5
Total-etching, Rebilda DC, standard mode	TRS	10	27.0[d]	4.7
Self-etching, Rebilda DC, standard mode	SRS	10	23.7[b,c,d]	3.1
Total-etching, Build-It FR, exponential mode	TBE	10	26.0[c,d]	4.5
Self-etching, Build-It FR, exponential mode	SBE	10	23.8[b,c,d]	4.8
Total-etching, Rebilda DC, exponential mode	TRE	10	26.7[c,d]	4.4
Self-etching, Rebilda DC, exponential mode	SRE	10	24.8[b,c,d]	4.4

* Values designated by the same superscript letter are not statistically different at p>0.05

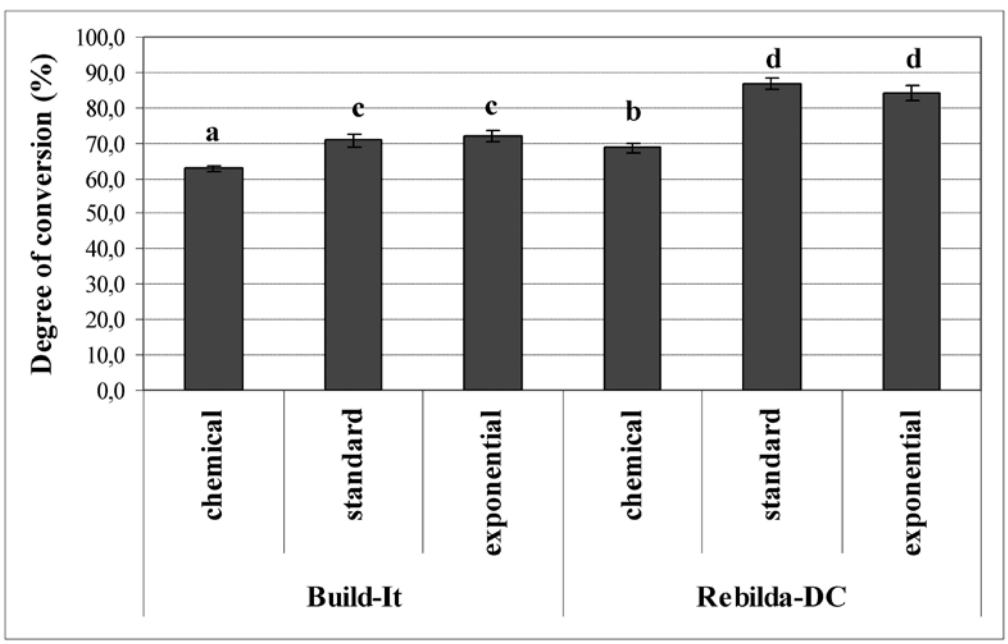
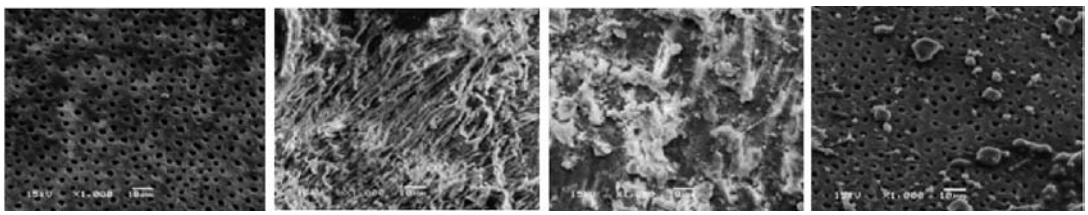


Fig. 3 Mean degree of conversion (DC%) values and standard deviations of the test materials at 15 min post-polymerization. Values designated by the same superscript letter are not statistically different at p>0.05.



Grope code	N	Adhesional between resin core and dentin	Cohesional within dentin	Cohesional within resin core	Mixed
TBC	10	8	0	2	0
SBC	10	6	1	2	1
TRC	10	8	0	1	1
SRC	10	7	1	1	1
TBS	10	8	0	0	2
SBS	10	8	0	0	2
TRS	10	9	0	0	1
SRS	10	8	0	0	2
TBE	10	10	0	0	0
SBE	10	8	0	1	1
TRE	10	10	0	0	0
SRE	10	9	0	0	1

Fig. 4 Failure mode analysis of test groups. SEM figures above show representative images for each failure mode. Original magnification ×1000, bar=10 μm.

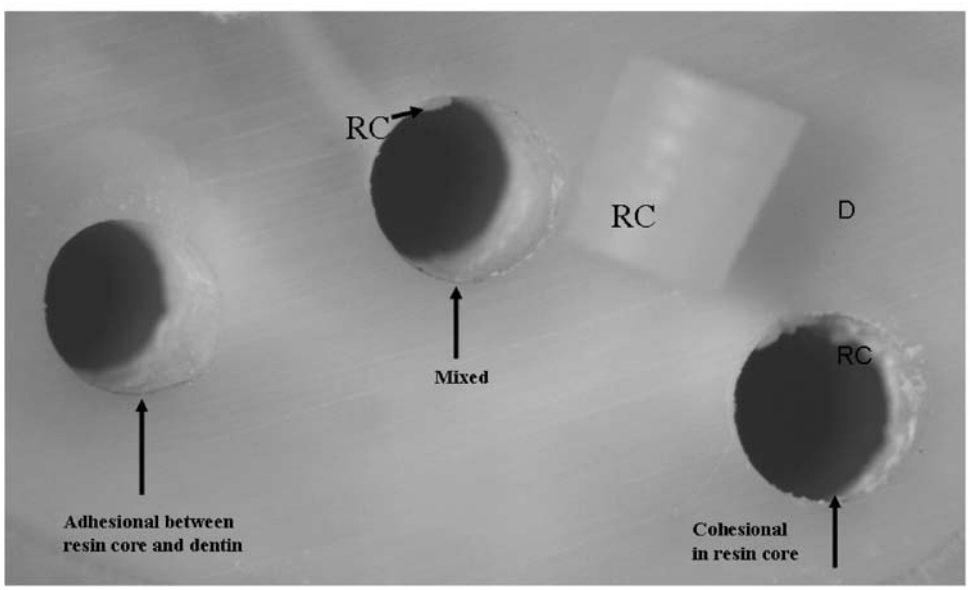


Fig. 5 Stereomicroscopic images of failure types. RC: resin composite, D: dentin (×40 magnification).

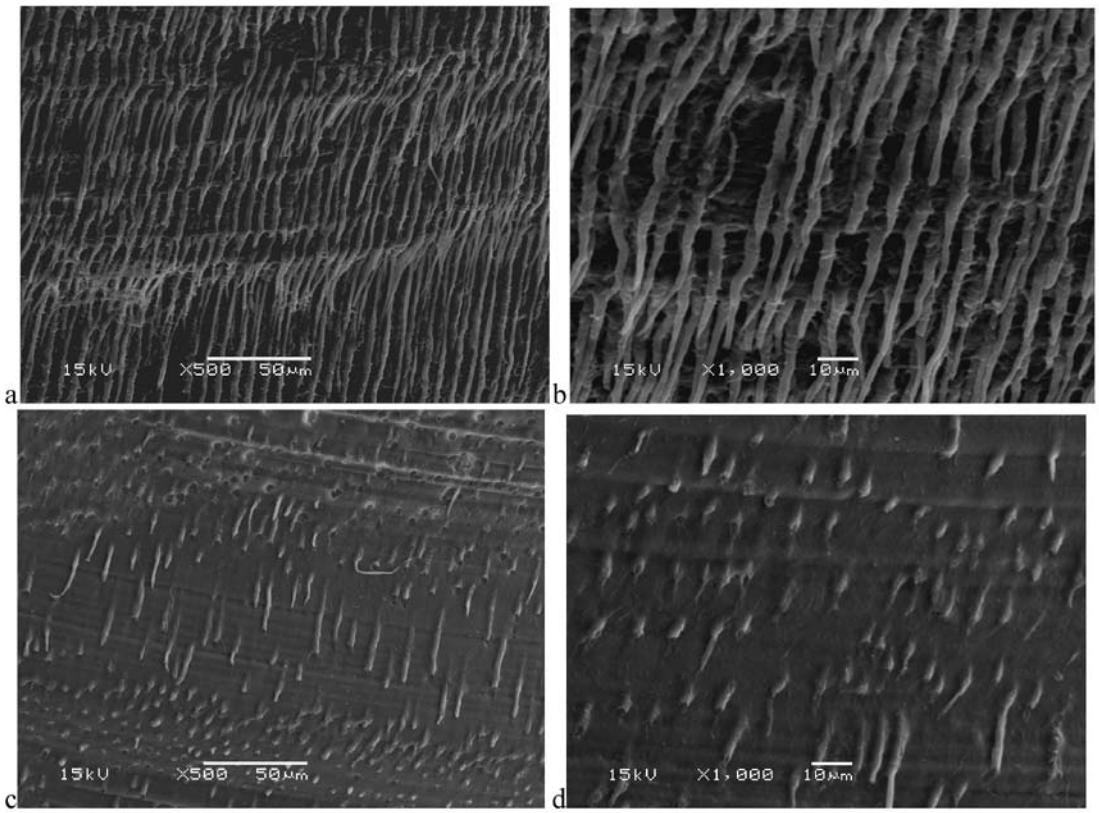


Fig. 6 SEM photomicrographs of the resin tag system after dissolving tooth structure with 2 M HCl. With Adper Scotchbond Multi-Purpose adhesive system, resin replica of the dentin surface revealed a regular tag distribution with deep penetration and a dense and fibrous structure: (a) original magnification×500, bar=50 μm; (b) original magnification×1000, bar=10 μm. With Clearfil SE Bond adhesive system, porous and thin resin tags extended longitudinally to dentin wall. In many areas, there were no evidences of tag formation and when tags were present, they had irregular shapes: (c) original magnification×500, bar=50 μm; (d) original magnification×1000, bar=10 μm.

with LED standard and exponential modes ($p<0.05$). At 15 minutes after 20-second radiation in LED exponential mode, Build-It FR showed 72% for DC whereas Rebilda DC showed 84.2%. When polymerized in LED standard mode, Rebilda showed the highest DC value at 86.8±1.6%.

Figure 4 shows the distribution of failure modes and the corresponding SEM photomicrographs. Adhesive failure at the interface between resin core and dentin accounted for most of the failures of the test groups. Very few specimens exhibited cohesive failures in dentin or composite, and mixed failures. Figure 5 then shows the stereomicroscopic images of adhesive failure at dentin-composite interface, mixed and cohesive failures in resin core. Figure 6 shows the SEM photomicrographs of the tooth structure after demineralization with HCl.

DISCUSSION

The μ-PO bond strength test used in this study was a modification of a previous test set-up[25], and this test method offered us the opportunity to test five specimens from one disk. It is worthwhile to point out that some standard deviations in push-out bond strength values were observed, which could be attributed to the lower values obtained from the cavities in the middle of each dentin disk in all the groups. It is known that some intrinsic factors such as regional structural differences, pulpal pressure, dentin permeability, water content, and surface morphology have effects on dentin bond strength[26]. Further on the influence of test methods, previous studies conducted using conventional test methods such as shear and tensile testing, followed by failure analysis, revealed that a large number of specimens fractured cohesively within the resin composite[14]. By contrast, in the present experiment which used the μ-PO test method and failure analysis, it was

revealed that each group with respect to resin core system, adhesive system, and polymerization mode predominantly showed adhesive failure between resin composite and dentin (Fig. 4).

To minimize shrinkage stress, "soft cure" or "soft-start" light curing units were developed[19]. The "soft-start" method employs an initial light activation at low irradiance followed by a second at a higher irradiance that is typically equivalent to that used in the continuous curing method[27]. In this study, when comparing the effect of different polymerization modes of LED on both bond strength and degree of conversion, no significant differences were observed between exponential and standard modes. Similarly, a previous study by Rahiotis *et al*. which investigated the curing efficiency of different types of light curing units, they reported that no statistically significant differences were detected in degree of conversion between the two curing modes of Elipar TriLight unit (continuous *versus* exponential ramp)[28]. Further on the effect of initial light intensity, an investigation by Santos *et al*. showed that resin-dentin bond strengths yielded with initial light intensities of 300 mW/cm^2 and 150 mW/cm^2 in step-cure technique were not statistically different from those yielded with continuous exposure curing method[29].

Apart from resin-dentin bond strength evaluation, this study also used FT-IR spectroscopy with ATR sensor to evaluate the degree of conversion of different resin core systems produced by chemical polymerization *versus* the exponential and standard modes of LED. At this juncture, it must be put into perspective that it is difficult to compare DC values among different research studies, since the remaining double bonds may depend on several factors[30]. In the context of this study, chemically polymerized specimens presented statistically lower degree of conversion values in all the tested materials. This finding was in accordance with previous studies showing that dual-cured resins depended on light irradiation to achieve an optimal degree of conversion[31,32].

As for the comparison of DC values between the resin core systems, Build-It FR showed significantly lower DC values than Rebilda DC. This could be partially attributed to differences in filler load, filler type, resin matrix, and formulation. On this note, it is noteworthy that the ratio of filler relative to resin is important, since penetration of light into the composite is more difficult when the filler proportion is higher[19]. The amounts of fillers used in this study were 68.2% and 70.2% by mass for Build-It FR and Rebilda DC respectively. Although both resin core systems had similar filler loading, Rebilda DC showed a significantly higher degree of conversion than Build-It FR. This result was consistent with a previous study which reported on dual-cure luting resins with similar filler loading but with different degree of conversion values[31].

Build-It FR is a compound comprising bisphenol A-glycidyl dimethacrylate (Bis-GMA), difunctional monomers (urethane dimethacrylate (UDMA) and 1,6-hexanediol dimethacrylate (HDDMA)), while inorganic filler particles are silane-treated glass fillers and chopped glass fibers. As for Rebilda DC resin core system, it has a different matrix composition comprising Bis-GMA, UDMA, and triethyleneglycoldimethacrylate (TEGDMA) — which could have accounted for a higher degree of conversion. Indeed, a study by Trujillo-Lemon *et al*. showed that TEGDMA and HDDMA yielded DC values of 83±1% and 76 ± 2% respectively[33]. In addition, the higher degree of conversion of Rebilda DC might be related with its lower viscosity. Lower viscosity results in greater DC% increasing the mobility of molecules[24].

This study also compared the effects of three-step total-etch technique against two-step self-etch technique. It was found that the total-etch adhesive system exhibited higher bond strength values than the self-etch adhesive system. These results might indicate that the penetration ability of Clearfil SE Bond to dentin was lower than that of Adper Scotchbond Multi-Purpose adhesive system. With two-step self-etch adhesives, they are based on the separate application of self-etch primer and hydrophobic resin, whereby mild self-etch primers are typically used. Mild self-etch adhesive systems (pH<2) are able to partially remove the smear layer and penetrate the dentinal surface, creating less pronounced resin tag formation and thinner hybrid layers than those of total-etch systems[8]. Our SEM observations confirmed the previous findings[34], whereby specimens prepared from the two different adhesive systems showed differences in surface texture.

With Adper Scotchbond Multi-Purpose (Figs. 6a and 6b), resin tags were easily observed. Conversely, with Clearfil SE Bond (Figs. 6c and 6d), a typically thin hybrid layer was shown coupled with an absence of resin tags. This, therefore, might suggest a lower penetration ability of the primer into the tubules. It should also be noted that freeze-drying method was used in this study to reveal the resin tag system. This method was based on sublimation of water from ice in low pressure to avoid structural collapse caused by capillary forces. It has been used previously with success for dentin and enamel[23]. However, it should also be mentioned that none of the drying methods to-date could sustain the original structure of the resin tag system. This then partly explained the surface structure of the self-etching system observed in this study.

Recently, it has been demonstrated that self-etch adhesive systems were incompatible with

chemical-cured composites because of the presence of activator[35]. In the present study, the lowest values for bond strength were recorded for chemically polymerized Build-It FR with Clearfil SE Bond, thereby illustrating and confirming the incompatibility of this combination. However, the bond strength of Rebilda DC was not affected by the self-etch adhesive system.

The ideal build-up material is the natural tooth structure. On this score, it is advisable to preserve as much healthy tooth structure as possible. Further, with the use of a suitable dentin bonding system, the superior adhesive bonding between the build-up material and tooth is an unbeatable merit[1]. In the context of the present study, its results suggested that further long-term clinical trials are needed to clarify the influences of bond strength and degree of conversion on the longevity of resin core systems.

CONCLUSIONS

Within the limitations of this *in vitro* study, it could be concluded that:

1. Dual-cure resin core systems showed different bond strengths depending on their composition, polymerization mode, and the adhesive system used;
2. Polymerization mode might be important for the long-term clinical performance of restorations; and
3. Use of total-etch adhesive systems might be preferred for dual-cure resin core build-ups than self-etch adhesive systems.

ACKNOWLEDGEMENTS

This study was financially supported by the Scientific Research Projects Fund of Gazi University and a scholarship grant from the Center for International Mobility. This work was also supported in part by a contribution from the Bio- and Nanopolymers Research Group, Center of Excellence of the Academy of Finland.

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