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The Effect of Thermal Stress on Bonding Durability of Resin Composite Adaptation to the Cavity Wall

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This study evaluated the effect of thermal stress on marginal sealing and cavity wall adaptation using two adhesive systems. Cylindrical cavities were prepared in superficial dentin of bovine incisors and bonded with Clearfil SE Bond or Single Bond adhesive. Cavities were bulk-filled with Photo Clearfil Bright or Filtek Flow resin composite and light-cured for 40 seconds. Specimens were thermocycled for 0, 500, or 5000 times. A dye penetration test was carried out to determine adaptation to the cavity wall. Dye penetration length was calculated as a percentage of the total cavity wall length. Clearfil SE Bond showed excellent marginal sealing and cavity wall adaptation regardless of composite type up to 500 cycles of thermal stress. As for the Single Bond groups, significantly greater marginal leakage occurred after 500 cycles. At 5000 cycles of thermal stress, both adhesive systems showed significantly decreased marginal integrity compared with the 0 cycle group.

Keywords: Thermal stress, Bond durability, Adaptation

INTRODUCTION

Resin composites undergo polymerization contraction — which generates stress within the resin composite and also at the restoration-tooth interface. This stress has been shown to lead to gap formation between the resin and cavity surface[1]. Such marginal gaps and subsequent microleakage may lead to marginal staining[2], postoperative sensitivity[3,4], and secondary caries[5].

To enhance bond integrity between a resin composite restoration and the cavity preparation, bonding agents are used. If the adherence mediated by the adhesive system is successful against the polymerization contraction stresses, the bond will survive. On this note, most contemporary adhesive systems render relatively favorable bonding effectiveness immediately after light curing, regardless of the adhesive used[6,7]. At this juncture, it should be highlighted that clinically, bonded restorations are subjected to repeated thermomechanical stresses that create additional stresses on the bond during the life of a restoration. As a result, deterioration of the adhesive bonding at the resin-tooth interface may occur at the margin of a restoration and also along the cavity wall.

Bonding durability is an important factor for a long-lasting bonded restoration clinically. Although a long-term clinical trial is considered the ideal method to confirm the quality of a restorative system, it is difficult to discriminate among — as well as control — the various factors that can cause a restoration to fail[8]. Furthermore, clinical trials are costly and time-consuming. Therefore, laboratory research is frequently performed to examine the qualities of dental restorative materials. To assess the long-term effectiveness of bonded restorations, many laboratory studies are carried out that include the evaluation of bond strength, fracture toughness, leakage, and marginal or cavity wall integrity[8]. To simulate the long-term effects in an oral environment, an artificial degradation technique is typically applied to the specimens. One of the widely used degradation simulation techniques is the thermocycling test. It has been demonstrated that increased destruction occurs to bonds between tooth substrates and resin composite restorations after thermocycling[9,10].

Presently, hybrid resin composites are widely accepted and regarded as a universal dental resin-based restorative material for direct-bonded restorations in any part of the oral cavity. As for flowable resin composites, they are modified resin-based restorative materials that are available as a low-viscosity filling material and which can be placed in small cavities or as a surface coating[11]. The more highly filled hybrid composites exhibit low shrinkage and a high elastic modulus[12]. In contrast, flowable composites have a reduced filler load that results in greater shrinkage, but a lower shrinkage stress and decreased elastic modulus[13]. The latter two characteristics might be less detrimental to the tooth-resin interface with respect to gap formation[12]. However, the reduced mechanical properties of flowable composites may influence the clinical performance of restorations over the long term.

The purpose of this study was to evaluate the effect of thermal stress on marginal sealing and cavity wall adaptation of two types of resin composite (flowable *versus* hybrid material) with two different bonding systems (self-etching filled adhesive *versus* total-etch unfilled adhesive). The null hypotheses were: (1) thermal stress will not affect marginal sealing and cavity wall adaptation, and (2) neither of the bonding resins or resin composites used will affect marginal sealing or cavity wall adaptation.

MATERIALS AND METHODS

Specimen preparation

Tables 1 and 2 list the materials, components, manufacturers, and batch numbers used in this study. Forty-eight erupted intact bovine lower incisors stored frozen immediately after extraction were used.

Labial enamel was removed using a model trimmer under running water to expose a flat superficial dentin surface, and finished with wet 600-grit silicon carbide paper. Two cylindrical cavities, 1 mm deep and 3 mm in diameter with a C-factor of 2.3, were prepared on the flat dentin surface of each tooth using 100-μm-grit diamond points (#CR30, ISO 068 029, GC, Tokyo, Japan; #430, ISO 041 019, Shofu, Kyoto, Japan) with copious air-water spray. Four teeth were allocated to each of the 12 groups (Fig. 1).

The teeth were randomly assigned to one of the two adhesives used: self-etching priming system — Clearfil SE Bond (Kuraray Medical, Tokyo, Japan) or phosphoric acid etching system — Single Bond (3M ESPE, St Paul, MN, USA) (wet bonding technique). After light curing of the adhesive for 10 seconds, the cavities were bulk-filled with Photo Clearfil Bright (Shade US, Kuraray Medical) or Filtek Flow (Shade A3, 3M ESPE) resin composite. The materials were polymerized using a light intensity of 600 mW/cm^2 for 40 seconds. The light curing unit used was an experimental quartz-halogen light curing unit (GC). Distance between light tip and resin surface was less than 1 mm, and intensity at the top of the specimens was measured with a curing radiometer (Model 100, Demetron Research Corp., Danbury, CT, USA). After light curing was completed, the specimens were stored in the dark for 24 hours in 37℃ water. All restorations were finished with 600-grit silicon carbide paper under running water to expose the tooth-restoration interface and to ensure that no flash remained at the margins.

Thermocycling procedure

Before thermocycling, root apices were sealed using Clearfil SE Bond and Photo Clearfil Bright composite. The external tooth surface surrounding the restoration in each tooth was coated with nail varnish except for a 1-mm area around the restoration margin. Specimens for each bonding system and each composite were randomly divided into three thermocycling subgroups with four specimens per

Table 1 Bonding systems used in this study

Material/Manufacturer	Batch No.		Components	Bonding procedure
Clearfil SE Bond (Kuraray Medical, Tokyo, Japan)	Primer:	00316A	MDP, HEMA, dimethacrylates, photoinitiator, water	a (20s) , b, c, d, e (10s)
	Bond:	00403A	MDP, HEMA, Bis-GMA, dimethacrylates, photoinitiator, microfiller	
Single Bond (3M ESPE, MN, USA)	Etchant:	3GP	35% phosphoric acid	f (15s), g, h, c, d, e (10s)
	Bonding	3HX	Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer, photoinitiator, ethanol, water	

MDP: 10-methacryloyloxydecyl dihydrogen phosphate
HEMA: 2-hydroxyethyl methacrylate
Bis-GMA: bisphenol A diglycidylmethacrylate
Procedure steps: (a) active priming; (b) gently air-dry; (c) apply adhesive; (d) gently air-blow; (e) light-cure; (f) acid-etch; (g) rinse with water; (h) blot dry.

Table 2 Resin composites used in this study

Material/Manufacturer	Code	Components	Filler Load		Batch No.
Photo Clearfil Bright (Kuraray Medical, Tokyo, Japan)	PB	Bis-GMA, dimethacrylates, silanated colloidal silica, prepolymerized organic filler containing colloidal silica, photoinitiator	Weight %:	82	00040B
Filtek Flow (3M ESPE, MN, USA)	FF	Bis-GMA, TEGDMA, dimethacrylate polymer, zirconia/silica filler, photoinitiator	Weight %: Volume %:	68 47	2CY

Bis-GMA: bisphenol A diglycidylmethacrylate
TEGDMA: triethylene glycol dimethacrylate

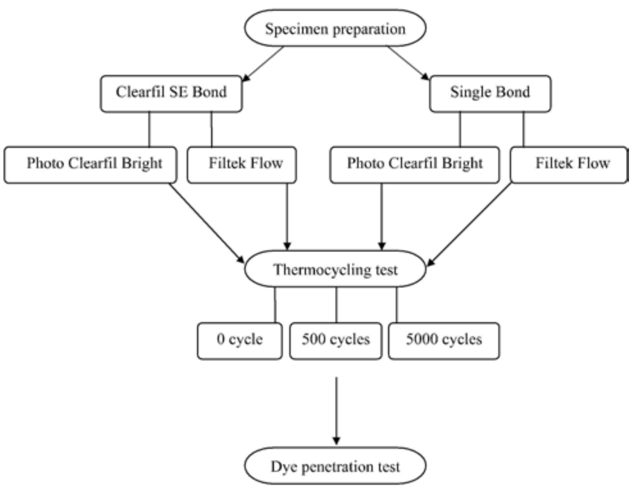

Fig. 1 Diagrammatic representation of the experimental design.

subgroup. The designated number of cycles for thermal stressing were 0, 500, and 5000 cycles. The first subgroup of 0 cycle was a control group. The other two subgroups were subjected to the thermal cycling test in water baths between 5°C and 55°C with a 30-second dwell time in each bath.

Evaluation of marginal sealing and cavity-wall adaptation

To determine the degree of adaptation to the cavity margins and walls, a dye penetration test was performed. The dye penetration test consisted of placing 1.0% acid red in propylene glycol solution (Caries Detector, Kuraray Medical) on the margin of the restorations for five seconds, followed by rinsing with water and gentle air-drying.

Degree of dye penetration was observed using a stereomicroscope at X20 magnification. A photographic record of each specimen was obtained at this stage. Specimens were cut in half longitudinally using a slow-speed diamond saw (Isomet, Buehler, Lake Bluff, IL, USA), and the dye was re-applied to the cavity walls and observed to identify the presence of gaps and photographed.

From the photographs, the length of dye penetration along the cavity margins and cavity walls was measured using a digitizer (model KD4300, Graphtec, Tokyo, Japan). Degree of marginal sealing was determined as the ratio of the margin stained with dye divided by the total length of the margin and then converted to a percentage. Dye penetration along the cavity walls was calculated as a percentage of the total cavity wall length. This was referred to as cavity-wall gap formation. Dye penetration scores were compared and analyzed using the Kruskal-Wallis test and Mann-Whitney U test at $\alpha=0.05$.

RESULTS

Table 3 shows the results of marginal gap formation. Marginal gap formation significantly increased after 5000 thermal cycles regardless of adhesive system and composite type ($p<0.05$). With Single Bond, 500 cycles showed a significant increase in marginal gap formation ($p<0.05$). With Clearfil SE Bond, the 5000-cycle group showed significantly greater marginal gap formation compared with the 500-cycle group for both resin composites ($p<0.05$). In the Single Bond groups, significantly greater marginal gap formation was observed when compared with Clearfil SE Bond groups regardless of composite type at 500 and 5000 cycles ($p<0.05$), except for cavities restored with Photo Clearfil Bright after 5000 thermocycles.

Table 4 shows the results of cavity-wall gap formation. Cavity-wall gap formation significantly increased after 5000 thermocycles for Filtek Flow composite with both adhesive systems ($p<0.05$). With

Table 3 Degree of marginal gap formation as a percentage (% mean (SD))

Number of thermocycles	Marginal gap formation (%)			
	Clearfil SE Bond		Single Bond	
	PB	FF	PB	FF
0	0	0	0.3 (1.0)	0.3 (0.7)
500	1.8 (3.4)[a]	0[b]	12.3 (5.5)[a]	16.0 (7.0)[b]
5000	12.1 (5.8)	10.2 (6.1) c	15.9 (6.7)	19.9 (6.9)[c]

Same superscript letters and vertical lines indicate statistically significant differences (p<0.05).

Table 4 Degree of cavity-wall gap formation as a percentage (% mean (SD))

Number of thermocycles	Cavity-wall gap formation (%)			
	Clearfil SE Bond		Single Bond	
	PB	FF	PB	FF
0	12.9 (8.5)[d]	0[d]	13.1 (11.6)	0.6 (0.9)
500	15.3 (8.3)[e]	4.5 (7.7)[e, f]	18.0 (5.0)	18.4 (4.1)[f]
5000	17.0 (7.3)	17.5 (5.6)	22.9 (7.4)	23.2 (7.9)

Same superscript letters and vertical lines indicate statistically significant differences (p<0.05).

Photo Clearfil Bright, there were no significant differences in cavity-wall gap formation for Single Bond at any thermocycling regime (p>0.05). Clearfil SE Bond combined with Filtek Flow showed significantly better adaptation to the cavity wall as compared to Photo Clearfil Bright groups at 0 and 500 cycles (p<0.05). Single Bond showed significantly greater cavity-wall gap formation compared with Clearfil SE Bond for Filtek Flow restorations under the 500-cycle condition (p<0.05). No significant differences in cavity-wall gap formation were observed among the experimental groups after 5000 thermocycles, regardless of resin composite type and adhesive system used (p>0.05).

DISCUSSION

From the results of this study, both hypotheses were rejected. Thermal stress affected both marginal sealing and cavity wall adaptation of the resin composite restorations. Significant differences in marginal sealing and cavity wall adaptation were found among the different bonding resins and resin composite materials examined.

Single Bond showed significant deterioration of the interfacial bond after 500 thermocycles, whereas the bond integrity of Clearfil SE Bond deteriorated at 5000 thermocycles. However, the degree of marginal gap and cavity wall gap formation of Clearfil SE Bond groups were less than all Single Bond groups regardless of thermocycling regime. This result suggested that the thermal susceptibility of bonded restorations varied with the adhesive system employed[14,15]. This might be due to the different bonding mechanisms between total-etch adhesive system and self-etching primer adhesive system. After acid etching with phosphoric acid, the bonding resin might not be able to penetrate to the base of the demineralized dentin layer. As a result, the presence of unprotected demineralized collagen fibers led to early bond degradation[16].

The mechanical properties of each component of resin-dentin bond complex, *i.e.*, resin composite, adhesive layer, hybrid layer, and underlying dentin, are believed to play significant roles in bond stability that might contribute to bond durability[17,18]. A recent study on water sorption and solubility of various bonding agents was performed by using resin disks before and after water immersion for three days[19]. It was revealed that Clearfil SE Bond had significantly less water sorption compared to the other commercial adhesives investigated: Excite, Scotchbond Multi-Purpose adhesive, One-Up Bond F, and Xeno III[19]. During the thermocycling procedure, the specimens were subjected to temperature changes and water immersion. Lower water sorption, and hence less water molecules within the polymer matrix, helps to maintain the mechanical properties of dental resins over time.

In the current study, the bonding resin of the self-etching primer system absorbed less water[19]. This lower water sorption behavior might thus account for the more durable bond as compared to the adhesive system based on phosphoric acid etching — a result consistent with previous studies that evaluated and compared the bond strength between these two adhesive systems[20-22].

This study demonstrated better cavity wall adaptation with Filtek Flow flowable composite as compared to Photo Clearfil Bright hybrid composite for non-thermocycled teeth[23,24]. However, after 5000 thermal cycles, the flowable composite showed increased gap formation at the bonded interface, whereas the hybrid composite showed no significant changes in gap formation between 0 and 5000 cycles. Mean gap values were no different after 5000 thermocycles, regardless of which resin composite or bonding system being used.

Filler content in resin composites has an inverse correlation to the linear coefficient of thermal expansion[25,26]. This physical property has been demonstrated to be related to microleakage in resin-based restorations[27]. After a composite restoration is exposed to thermal stress for a long period of time, the greater coefficient of thermal expansion of the flowable composite may result in increased gap formation at the resin-dentin interface. Hence, thermal stress is believed to be an important factor that influences bond durability[20], especially for cavities restored with flowable composite resins[28]. In this study, although the cavity wall adaptation of hybrid composite restorations was more stable when exposed to thermal stress, there were no differences in mean gap formation when compared to flowable composite restorations. In other words, stress effect may act only on marginal integrity of resin composite restorations. These findings were in agreement with a study which reported that a flowable composite showed significantly more microleakage than hybrid composite restoration after thermocycling[28].

Cavities restored with Clearfil SE Bond and Filtek Flow showed excellent marginal sealing and cavity wall adaptation in the control group (no thermal cycling). Moreover, 500 thermocycles did not significantly damage the bond along the tooth cavity. This could be attributed to the lower modulus of elasticity of flowable resin composites[12] in comparison with hybrid composites. As a result, a slightly more flexible Filtek Flow filling material at the bonded interface served to resist the stresses from polymerization shrinkage and thermal changes. In addition, a thick adhesive layer and well-formed resin-impregnated dentin layer created by Clearfil SE Bond could be responsible for good adhesion between the tooth substrate and restoration[29].

However, 5000 thermal cycles significantly affected the adaptation of flowable composite restorations. It could be that 5000 thermocycles could generate sufficient stress and movement within the materials such that the initial advantageous effects of flowable composite and that of a thick adhesive layer were overcome. As a result, increased gaps formed at the tooth-composite interface. Therefore, the use of 5000 thermocycles could be important in accelerating the ageing effect on tooth-resin interface. This study showed significantly less marginal gap formation for flowable composite restoration in association with Clearfil SE Bond than with Single Bond. Furthermore, for Filtek Flow restoration, Clearfil SE Bond initially showed distinctly better cavity wall adaptation than Single Bond, although no significant differences were found between the two groups after 5000 cycles. Therefore, self-etching adhesive systems — such as Clearfil SE Bond — seemed to be more promising in maintaining a more durable restoration.

In this study, 500 thermocycles showed variations in interfacial bond adaptation depending on the adhesive system used. The 500-cycle regime used followed the guidelines of ISO TR 11405, which proposed thermocycling test to accelerate the ageing of restorations[30]. However, results of the current study showed that the resin-dentin bond was significantly damaged only after 5000 cycles, when compared with the non-thermocycled group. This result showed the effect of thermal cycling on resin-dentin bonding: the latter was dependent not only on the number of thermal cycles, but also the adhesive system and resin composite used[14,15,28].

CONCLUSIONS

From the outcomes of this work, it would seem that when compared to the total-etch, unfilled adhesive system of Single Bond, the self-etch, filled adhesive system of Clearfil SE Bond was more superior in attaining a more reliable seal and better adaptation of resin composite to the cavity wall. This bonding and sealing performance of Clearfil SE Bond was exhibited regardless of composite type under thermal stress conditions.

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