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ORIGINAL ARTICLE

The effect of LED curing mode on microleakage of Class V cavity restored by silorane-based composite

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Abstract

Objective. This *in vitro* study evaluated the effect of soft-start curing mode by LED unit on the marginal microleakage of silorane composite restoration. ***Materials and methods.*** Class V cavities were prepared on the buccal surfaces of 80 extracted molars at the cementoenamel junction and randomly divided into eight groups. In groups 1–4, four adhesive/composite combinations (silorane/silorane, silorane/Z250, Adper Single Bond/Z250, Clearfil SE Bond/Clearfil AP-X) were applied and the composites were cured under standard mode at 1500 mW/cm^2 for 20 s. In the other four groups, after applying the same adhesive/composite combinations, the composites were cured at 550 mW/cm^2 for 10 s followed at 1500 mW/cm^2 for 15 s. After 24 h of water storage and thermocycling, the specimens were placed in 1% methylene blue solution. The dye penetration was assessed under a stereomicroscope. The data were analysed using non-parametric tests. ***Results.*** There were no significant differences among four groups for two curing modes at the occlusal and gingival margins ($p > 0.05$). The soft-start curing had a positive effect on the gingival marginal sealing of group 1 ($p < 0.05$), but not for the other three groups ($p > 0.05$). ***Conclusion.*** The beneficial effect of relatively high soft-start curing on marginal sealing of Class V restoration depends on a combination of the adhesive and composite types, having a positive effect on silorane adhesive/silorane-based composite at the gingival margin.

Key Words: *silorane composite, marginal sealing, curing mode, adhesive system*

Introduction

Despite increasing advances in adhesive resin systems, polymerization shrinkage of resin composites has remained a major problem to achieve a durable successful resin restoration. During free radical polymerization, conversion of the monomers to packed polymer network is responsible for the shrinkage [1,2]. The resultant shrinkage stress can lead to deleterious effects on early developing of bonding interface, tooth structure and restoration [3]. Microleakage, post-operative sensitivity and recurrent caries can be the clinical problems following failure at the adhesive interface [4,5].

Based on the destructive impacts of polymerization stress on clinical performance of composite restorations, considerable attempts have been made to minimize the stress. To achieve this purpose, composite placement in layering techniques [6], use of a low-modulus resin layer [7] and soft-start curing have been proposed [4,5,8,9]. Recently, a novel resin composite has been introduced as a low shrinkage resin containing siloxane and oxirane functional moieties; in which cationic ring opening polymerization led to volumetric expansion. This mechanism accounts, to some extent, for compensating the shrinkage created by monomer packing/bonding [10,11].

Regarding the ongoing development of LED technology, this light-emitting diode curing unit has been widely employed in dental practice due to its advantages such as small, wireless design with stable power and spectra, higher curing efficiency and more life time with minimal heat generation compared to a halogen light curing one [12,13]. High intensity LED is capable of decreasing the time of curing, increasing the degree of conversion and depth of cure, particularly in deep cavities where the distance between the light curing tip and the most gingival layer of composite is unavoidable. However, a high intensity can also produce a higher polymerization

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Table I. Adhesive systems used in this study.

Adhesive system	Composition/batch #	Application mode
Silorane adhesive system (3M ESPE, St Paul, MN)	Primer: phosphorylated methacrylates, vitrebond copolymer, Bis-GMA, HEMA, water, ethanol, silica filler, initiator, stabilizers/N213019 Bond: hydrophobic methacrylats, phosphorylated methacrylates, TEGDMA, silica filler, initiator, stabilizers/N213052	Apply primer for 15 s with gentle agitation, gently air thin. Light cure for 10 s. Apply Bond to the entire preparation, gently air thin. Light cure for 10 s.
Adper Single Bond (3M ESPE, St Paul, MN)	Bis-GMA, HEMA, dimethacrylates, polyalkenoic acid copolymer, initiators, water and ethanol/N227596	Apply two consecutive coats of the adhesive for 15 s with gentle agitation, gently air thin for 5 s. Light cure for 10 s.
Clearfil SE Bond (Kuraray, Okayama, Japan)	Primer: MDP, HEMA, Hydrophilic dimethacrylate, dl Camphorquinone, N, N diethanol-p-toluidine, water/00977A Bond: MDP, Bis-GMA, HEMA, hidrophylic dimethacrylate, dl camphorquinone, N, N diethanol-p-toluidine, silanated colloidal silica/01453A	Apply primer on the cavity walls, leave it for 20 s, then gently air blow it. Apply Bond to the entire surface of the cavity wall and uniform it with a gentle air stream. Light cure for 10 s.

stress at the adhesive interface [4,5]. Hence, the concept of soft-start polymerization is still valid for less deep cavities (2 mm) with modern high power LED units [14]. Initial curing at a low intensity and following curing by a final high power can provide more time for the composite to flow. The slow stiffness development in the curing composite results in a lowered polymerization stress compared to continuous standard mode of curing [1,9,15,16]. This effect may enhance the marginal integrity of composite restorations [4,5,8,9,17–21]. With respect to reduction volume shrinkage to below 1% in silorane composite compared with 1.7–3.5% in methacrylate one [11,22], it seems that the low shrinkage composite is less sensitive to curing mode with high power LED. On the other hand, a direct relationship between contraction stress and microleakage was found in Class V cavities [23,24]. Therefore, the effect of stepped soft-start curing in comparison with a standard high power one by LED on the marginal sealing of silorane and methacrylate composite restorations is an important clinical issue in shallow cavities such as Class V cavity. This effect was evaluated in the current study by a microleakage test with silorane and methacrylate composites associated with their respective adhesives, two-step self-etch and etch-and-rinse systems.

Materials and methods

Eighty extracted human molars without caries or restorations were collected and cleaned for this *in vitro* study. The teeth were stored in a 0.1% thymol solution no longer than 1 month after extraction. Box-shaped Class V cavities (5 mm wide, 3 mm high, 2 mm deep) with the gingival margin 1 mm below the cementoenamel junction were prepared on the facial surface of each tooth using fissure diamond burs (Teezkavan, Tehran, Iran) in an air/water cooled high speed turbine. The occlusal margins were located in the enamel and the gingival margins were located in the dentin. Each bur was exchanged following the preparation of the 10 cavities.

The prepared teeth were randomly divided into four groups of 20 teeth each according to the adhesive/composite combination used. Each group was further divided into two sub-groups according to the curing mode (n = 10).

- *Group 1* (silorane/silorane): Silorane adhesive system, self-etch adhesive (3M ESPE, St Paul, MN, USA) was applied on the cavity walls containing the enamel and dentin surfaces according to the manufacturers' instruction (Table I). The silorane composite (Filtek P90, 3M) was then inserted in the cavity in two increments.
- *Group 2* (silorane/Z250): The silorane adhesive system (3M) was applied on the cavity walls containing the enamel and dentin surfaces according to the manufacturers' instruction (Table I). The cavity was filled using the composite Z250 (3M) in two increments.
- *Group 3* (Adper Single Bond/Z250): The cavity surfaces were etched with 32% phosphoric acid gel (3M) and rinsed for 20 s and gently blown to remove the excess water. The Adper Single Bond adhesive system (3M) was applied on the cavity surfaces according to the manufacturers' instruction. Filling the cavity was done using Z250 in two increments.
- *Group 4* (Clearfil SE Bond/Clearfil AP-X): The Clearfil SE Bond, self-etch adhesive system (Kuraray, Japan) was applied on the cavity walls containing the enamel and dentin surfaces according to the manufacturers' instruction (Table I). The Clearfil AP-X composite (Kuraray, Tokyo, Japan) was placed in the cavity in two increments.

Table II. Microleakage scores obtained from standard curing mode.

		Occlusal margins					Gingival margins				
Group	Adhesive/Composite	0	1	2	3	4	0	1	2	3	4
1	Silorane/Silorane	7	2	1	0	0	2	5	1	1	1
2	Silorane/Z250	6	3	1	0	0	5	3	1	1	0
3	Adper Single Bond/Z250	8	1	1	0	0	2	4	4	0	0
4	Clearfil SE Bond/C.APX	7	2	1	0	0	2	6	1	1	0

In the four groups, curing the composite was performed under standard mode of a LED unit (radii plus, SDI, Australia) at light intensity of 1500 mW/cm^2 for 20 s. In the other four groups, the composite was initially cured at 550 mW/cm^2 for 10 s, with a 5-s interval and continued at 1500 mW/cm^2 for 15 (soft-start mode). The initial intensity was provided by a 20 mm distancing between the light tip and the resin composite [4]. The intensity was measured with a radiometer. This distance was standardized using an opaque ring spacer. The energy density was approximately similar for both curing modes. In all the groups, the adhesives were cured with the radii plus unit at 1500 mW/cm^2 light intensity for 10 seconds.

After storage for 24 h in distilled water at room temperature, all the specimens underwent 1000 thermal cycles between 5–55°C in water baths with a 30-second dwell time. Then the teeth were coated with two layers of nail polish except for 1 mm around the restorations and the apices of the teeth were sealed with sticky wax as well. The teeth were immersed in a 0.5% methylen blue dye solution for 24 h. They were then washed with water, blot-dried and sectioned in the baccolingual plane through the center of the restorations with a water-cooled diamond wheel saw (Leitz 1600, Wetzlar, Germany). The sections were blindly observed for dye penetration by two independent examiners under a stereomicroscope (Carl Ziess Inc, Oberkochen, Germany) at 20× magnification. The microleakage extents were scored for both the occlusal and gingival margins from 0–4 as follows: 0 = no dye penetration; 1 = dye penetration extending for less than or up to 1/3 of the cavity depth; 2 = dye penetration of more than 1/3 or up to 2/3 of the cavity depth; 3 = dye penetration more than 2/3 of the cavity depth; and 4 = dye penetration extending along the axial wall.

The results were analyzed using Kruskal-Wallis and Mann-Whitney U non-parametric tests at the $p < 0.05$ level of significance.

Results

Microleakage scores for the occlusal and gingival margins in standard and soft-start curing modes are presented in Tables II and III, respectively. The Kruskal-Wallis tests were used to compare four different combinations of adhesive/composite for standard curing and soft-start curing separately, revealing no significant difference at the gingival and occlusal margins ($p > 0.05$). In order to evaluate the effect of curing mode on the microleakage of each adhesive/composite combination, Mann-Whitney U-test was performed for both the occlusal and gingival margins. These pair-wise comparisons showed that there were no significant differences in microleakage between standard and soft-start curing modes for groups 2, 3 and 4 at both margins ($p > 0.05$). However, soft-start curing significantly decreased the microleakage of group 1, silorane adhesive/silorane composite only at the gingival margin ($p = 0.005$). Comparison of all occlusal vs gingival margins, pair-wise, was done using Wilcoxon signed-rank test; in all groups, the occlusal margins presented a lower amount of microleakage than the gingival margins. However, this difference was statistically significant only for groups 1–4 (standard curing).

Discussion

Polymerization shrinkage is still one of the major challenges to achieve the perfect seal in composite

Table III. Microleakage scores obtained from soft-start curing mode.

		Occlusal margins					Gingival margins				
Group	Adhesive/Composite	0	1	2	3	4	0	1	2	3	4
1	Silorane/Silorane	10	0	0	0	0	9	1	0	0	0
2	Silorane/Z250	8	2	0	0	0	5	4	1	0	0
3	Adper Single Bond/Z250	8	2	0	0	0	7	1	1	1	0
4	Clearfil SE Bond/C.APX	7	3	0	0	0	7	2	1	0	0

restorations. Although several studies reported that soft-start curing could result in a significant reduced shrinkage stress during polymerization [25,26], there are conflicting results regarding this positive effect on the marginal integrity. The results of the current study revealed that the soft-start curing had no effect on the marginal sealing of methacrylate-base composites. This finding is in agreement with the results of previous studies [2,27–31]. Also, some researchers found that polymerization stress was not significantly affected by soft-start curing [32,33]. However, some reports demonstrated the beneficial effect of two-step curing on the marginal integrity [4,5,8,9,17–21]. The divergent data might be a result of different curing conditions including light intensity and curing time used in the first step, final intensity, total energy curing, distance of the curing light tip from the curing composite, type of curing unit, type of cavity and its c-factor and thickness of applied composite.

Some authors confirmed the more important role of adhesive systems over different polymerization techniques in determination of marginal sealing [29,33]. The main difference in authors' viewpoints regarding soft-start curing is initial intensity and its exposure time. Some authors [4,5] have suggested that the initial intensities of 45%, 50% and 70% of the final ones in the range of 250, 315 and 380 mW/cm^2 can produce a favorable result. A light intensity lower than 280 mW/cm^2 is not capable of starting an adequate reaction and final high intensity resulted in immediate curing [2,4]. On the contrary, Lim et al. [9] have demonstrated that the initial curing should be started by the intensity being lower than 100 mW/cm^2 with an exposure time of less than 5–7 s. The advantageous effect of the pre-set step curing mode provided by a commercially available curing unit with initial intensities of 100, 150 mW/cm^2 on the marginal integrity was not supported by some studies [2,27]. On the other hand, Amaral et al. [29] found that soft-start curing at 75 and 190 mW/cm^2 did not reduce the microleakage on a Class II cavity compared to conventional curing at 560 or 810 mW/cm^2.

It seems that determination of the optimal initial intensity in connection with the final intensity is difficult because this intensity should be sufficiently high to start a polymerization reaction and adequately low to maintain the composite in a deformable state to relieve polymerization stress. Several reports demonstrated that polymerization shrinkage rapidly increases during the first 10 s of light activation accompanied with maximum rate of polymerization stress [5,16,26,34]. Thus, in the present study, 10 s at 550 mW/cm^2 was applied as a soft-start curing. This intensity corresponds to 37% of final intensity at 1500 mW/cm^2. This curing protocol did not enable one to improve significantly the sealing ability of three combinations of adhesive/methacrylate composite when compared to curing at the high intensity of 1500 mW/cm^2. One possible explanation could be that the initial intensity used was too high for soft curing or high final curing at 1500 mW/cm^2 resulted in an increased total polymerization shrinkage that offsets any beneficial effect of the initial curing at 550 mW/cm^2 [34].

In a recent study [35], the soft-start mode of LED (650/800) could diminish polymerization shrinkage compared to curing at 800 mW/cm^2. The obtained results showed that the relatively high soft-start mode used in the present study could lead to a significant improvement of gingival sealing ability of silorane adhesive/silorane composite, but not for the methacrylate composite. This difference may be attributed to different kinetics of the polymerization reaction of the two type composites. Silorane composites possess a slower polymerization reaction with a higher gel point (7.6 s) [22,36]. Min et al. [37] speculated that silorane composite has the highest potential for relief of stress by flow before the gel point. In a recent study [22], the soft-start polymerization was capable of delaying the gel point (11 s), resulting in a trend in a reduced shrinkage stress; the high intensity curing led to the higher shrinkage stress rate. On the other hand, curing of silorane composite requires higher intensity (at least 500 mW/cm^2) compared to that methacrylate composite and an upper limit of light intensity at ~ 1200 mW/cm^2 was suggested [22,38].

According to our results, silorane and methacrylate composites revealed approximately similar microleakage in Class V cavity. A direct correlation between polymerization stress and microleakage was verified in some studies [23,24]. However, it was considered that shrinkage stress is not an inherent property of the material and is not simply decreased by reduction of volumetric shrinkage value [39]. In fact, development of stress is determined by a complex phenomenon involving several factors such as material properties (monomer type, filler content, filler/matrix interaction and viscoelastic properties) and rate of polymerization. The latter is influenced by photoinitiator concentrations, reducing agent, inhibitor, molecular weight and reactivity of monomer. In addition to these, shrinkage strain rate, cavity configuration, compliance capacity of cavity wall and quality of the adhesive bond impacted the marginal adaptation of composite restoration [20,24,30,39,40].

In this way, literature regarding silorane composites is a controversial issue; some authors described low shrinkage stress [11,22,40] and reduced cuspal deflection [41], while others reported no improvement of microleakage/stress [24,39,42,43] or modest decreases in the adverse effect of shrinkage stress [41]. The excellent sealing ability of silorane composite was demonstrated when using experimental [44] or commercial [45] two-step self-etch silorane adhesive. A recent study [46] indicated a significantly higher marginal adaptation of silorene adhesive/silorane composite compared to a total–etch/methacrylate one. In these studies, curing was performed at 800 or 1000 mW/cm^2. In the current study, similar excellent results were obtained in group silorane adhesive/silorane composite cured at 550/1500 mW/cm^2. This result may be related to high bonding performance of this new silorane adhesive in the case of controlled shrinkage stress. The self-etching primer of this adhesive with pH of 2.7 has been recently claimed to create chemical bonding to the hydroxyapatite crystals [47]. This bonding may contribute to the sealing ability of the adhesive after thermocycling. The excellent marginal sealing of the silorane system after thermomechanical load cycling [48] or water aging [49] has been recently reported when curing was performed at 750 mW/cm^2 [48].

Among the four groups tested, silorane and methacrylate composites were used associated with the same silorane adhesive system in two groups to rule out the differences between adhesive systems; no significant difference was shown in microleakage. Also, two other adhesives/methacrylates revealed no significant difference. This indicated the similar bonding quality of the two-step etch-and-rinse and the self-etch adhesives.

Also, the compatibility of silorane adhesive with methacrylate composite was exhibited. The similar bonding ability of the two types of composites associated with the silorane adhesive was recently found by Van Ende et al.'s [50,51] studies. This compatibility was attributed to the methacrylate-base of the silorane adhesive bond hydrophobic layer. However, silorane composite should be solely used associated with a silorane adhesive system not with methacrylate adhesive systems. Further studies are required to confirm the results in laboratory conditions with more simulating intra-oral situations.

Conclusion

The relatively high soft-start mode of curing (550/1500 mW/cm^2) used in this *in vitro* study enhanced the gingival sealing ability of silorane adhesive/silorane composite while it had no effect on silorane adhesive or methacrylate adhesive/methacrylate composite. The use of silorane composite compared to methacrylate had no positive effect in terms of marginal microleakage in Class V restorations cured with a high intensity.

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