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Resin composite shrinkage and marginal adaptation with different pulse-delay light curing protocols

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The aims of this study were, first, to measure shrinkage induced by different methods of pulse-delay light curing and, second, to verify their influence on the marginal adaptation of class V restorations in enamel and dentin. Eight groups, comprising seven groups ($n = 6$) with different pulse-delay parameters and a control group, were compared for dynamic linear displacement and force by using a fine hybrid composite. Based on these results, the pulse-delay curing procedure with the lowest shrinkage force was chosen and tested against the control group with respect to marginal adaptation in class V restorations ($n = 8$) before and after simultaneous thermal and mechanical loading. Statistically significant differences between groups were found for both shrinkage properties tested, with one pulse-delay group giving the lowest overall shrinkage values. However, the percentages of 'continuous margin' of this group, and of the control before and after loading, were not significantly different in dentin, whereas a significantly lower percentage of 'continuous margin' was detected in the pulse-delay group in enamel after loading.

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Polymerization shrinkage, being in the range of 2–3%, represents one of the most critical properties of composite restorative materials used for direct restorative techniques (1) as it may deform cavity walls (2) and create gaps between the tooth and the restoration (3). This is why its reduction remains one of the major goals of modern adhesive restorative dentistry (4), even if the literature does not support a strong positive correlation between marginal deterioration or marginal gap width and recurrent caries (5). The shrinkage reduction potential by chemical and/or structural modification of methacrylate-based composite materials appears limited (4). New composite matrix formulations based on ring-opening monomers may reduce polymerization shrinkage in an important way, but these materials are still under development (6). This is why effort is being put into the reduction of polymerization shrinkage of currently used light-curing composites by modifying their clinical application techniques (7). One way of reducing polymerization shrinkage consists of the use of specific layering techniques or flexible adhesive layers (8, 9). Another way, sometimes in combination with the use of flexible adhesive layers, is the modification of light-curing protocols (7), such as the soft-start or ramped light polymerization (10), the variable light intensity protocol (11), the intermittent polymerization (12) or the pulse-delay polymerization (13), sometimes also called pulse

activation (13, 14). The idea behind pulse-delay polymerization is the introduction of a relaxation period between the first, short pulse of light energy and the final, continuing polymerization. The rationale for this technique is the observation that both the shrinkage force, 10 min after light activation (15), and the shrinkage force rate (16) [also called shrinkage progression (6)] are significantly reduced. Different durations of the three phases of the pulse-delay method – pulse, relaxation, and final polymerization – have been described previously in the literature (14–19), raising the question on the influence of the duration of these phases on shrinkage behavior. Furthermore, only limited information is available on the marginal adaptation of restorations placed with the help of the pulse-delay light curing technique (17, 18, 20).

The purpose of this study was therefore to compare seven variations of the pulse-delay light curing technique, where the pulse and the relaxation times were varied, with a control group for dynamic linear displacement induced by shrinkage and for shrinkage force. Based on these results, the pulse-delay light curing procedure with the lowest shrinkage properties was identified and, in the second part of the investigation, tested against the control group with respect to marginal adaptation of mixed class V restorations before and after simultaneous thermal and mechanical loading.

The two hypotheses tested were that there are significant differences in the shrinkage behavior among the groups, and that a significantly better marginal

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adaptation in enamel and dentin is present before and after loading in the least shrinking pulse-delay group in comparison with the control group.

Material and methods

Shrinkage

The linear displacement induced by polymerization shrinkage was measured with a custom-made device, described in detail by STAVRIDAKIS *et al.* (21). In brief, it consisted of a stable metal frame, upon which a thin aluminum platelet with a perpendicular diaphragm was loosely placed. The edge of the diaphragm extended into a recess in the infrared measuring sensor. A standardized amount of the restorative material (Tetric Ceram A2, Lot E12349; Ivoclar Vivadent, Schaan, Liechtenstein) was placed on the aluminum platelet with the aid of a cylindrical Teflon mould. The material was then carefully flattened by means of a glass plate. Polymerization of the composite was carried out according to the protocols described in Table 1, by using a halogen light curing unit (VIP; Bisco, Schaumburg, IL, USA) from a distance of 1 mm through the glass plate. The light output was preset with the lamp's internal radiometer and regularly controlled with an external radiometer (Curing Radiometer 300; Demetron Research, Danbury, CT, USA). The displacement of the aluminum platelet caused by polymerization shrinkage of the test material was detected by a temperature-compensated infrared sensor with an accuracy of 100 nm and a sampling frequency of 1 Hz. The data were recorded by means of an A/D converter using custom made software with a personal computer (Macintosh IIfx; Apple computer, Cupertino, CA, USA) and stored on its hard disc. Six measurements were carried out for each group and the mean values were reported.

Measurements of polymerization shrinkage force were performed with a measuring device that was also described in detail by STAVRIDAKIS *et al.* (21). In brief, the upper part of the apparatus consisted of a semirigid load cell (PM 11-K; Mettler, Greifensee, Switzerland), to which a metal cylinder was screwed to mimic the natural deformation of cavity walls. The cylinder was coated with a standardized amount of composite (Tetric Ceram A2; Ivoclar Vivadent), which was compressed at a thickness of 1.5 mm onto a glass plate attached to the base of the device. To improve adhesion, the surfaces of the metal cylinder and of the glass plate were sandblasted with 50 μm Al$_2$O$_3$ (Microetcher; Danville Engineering, Danville, CA, USA) and silanized (Monobond S; Ivoclar Vivadedent). Light curing of the composite was

carried out, according to Table 1, by using the VIP halogen light curing unit (Bisco), through the glass plate at a distance of 1 mm from the test material. The force that built up during polymerization shrinkage was detected by means of the load cell at a sampling frequency of 1 Hz. The data were transferred real-time into the attached computer (Macintosh IIfx; Apple computer) via an A/D converter using custom-made software. Six measurements were carried out for each material, and their mean values were reported. The differences between groups were tested by using analysis of variance (ANOVA) and Tukey-Kramer Multiple Comparison Test with significance at a *P*-value of < 0.05.

Marginal adaptation

Eight intact, caries-free human upper molars with completed root formation were prepared for the experiments. They had been stored in 0.1% (v/v) thymol solution for the time between extraction and use in this *in vitro* test. After scaling and pumicing, the teeth were randomly assigned to the two experimental groups and mounted on custom-made specimen holders, with their roots in the center, using a cold-polymerizing resin (Paladur; Heraeus-Kulzer, Wehrheim, Germany).

One standardized V-shaped Class V cavity was cut orally and facially in each of the test teeth, with 50% of the margins located in enamel and 50% in dentin. To that purpose, 80-μm diamond burs (Universal Prep Set; Intensiv, Lugano, Switzerland) in a red contra-angle (IntraMatic 25CH; KaVo, Biberach, Germany) were used under continuous water cooling. The dimensions of the cavities were as follows: 3.0–3.5 mm in a horizontal direction, 2.5–3.0 mm in a vertical direction and a maximum depth of 1.5 mm. The margin in enamel was bevelled to a crescent-shape with a maximum width of 1.2 mm. The entire cavity was finished using 25-μm finishing diamond burs (Universal Prep Set; Intensiv). The cavity preparations were checked for marginal imperfections, such as fractures or chipping, under a stereo microscope (Wild M5; Wild Heerbrugg, Switzerland) at ×12 magnification, and corrected if necessary.

Enamel margins were etched with 35% H$_3$PO$_4$ gel (Ultra-Etch, Batch J016; Ultradent Products, Salt Lake City, UT, USA) for 40 s and the etching gel was subsequently rinsed with a copious amount of water spray for 30 s. After slight drying of the cavities with oil-free compressed air, Syntac Primer (Lot E52572; Ivoclar Vivadent) was applied on the entire cavity surface for 20 s and air-dried, followed by the application of Syntac Adhesive (Lot 08386; Ivoclar Vivadent) for 20 s and air-drying. Finally, Heliobond (Lot E10061; Ivoclar Vivadent) was applied onto the entire cavity surface, left to penetrate for 20 s, blown out into a thin layer by using slight stream of compressed air, and light-cured using a VIP halogen light curing unit (Bisco) at 600 mW cm^{-2} for 30 s. Composite (Tetric Ceram A2, Lot 12349; Ivoclar Vivadent) was applied in two increments, the first layer being placed in the cervical half of the cavity and the second layer in the occlusal half of the cavity. Each layer was light-cured using the VIP light curing unit (Bisco) according to the procedures of group 1 and group 8 (Table 1).

Immediately after polymerization, the restorations were finished and polished by using flexible discs with different grain sizes (SofLex PopOn; 3M Espe, Seefeld, Germany). Polishing was controlled using ×12 magnification under a stereo microscope and corrected if necessary. Subsequently, impressions were carried out of each restoration using a polyvinyl-siloxane impression material (President light body; Coltène-Whaledent, Altstätten, Switzerland).

Table 1

Description of the experimental groups for shrinkage measurements

Group	First light application	Relaxation	Second light application
1 (control)	–	–	10 s @600 mW cm^{-2}
2	1 s @400 mW cm^{-2}	5.0 min	10 s @600 mW cm^{-2}
3	2 s @400 mW cm^{-2}	5.0 min	10 s @600 mW cm^{-2}
4	3 s @400 mW cm^{-2}	5.0 min	10 s @600 mW cm^{-2}
5	2 s @400 mW cm^{-2}	0.5 min	10 s @600 mW cm^{-2}
6	2 s @400 mW cm^{-2}	1.0 min	10 s @600 mW cm^{-2}
7	2 s @400 mW cm^{-2}	3.0 min	10 s @600 mW cm^{-2}
8	2 s @400 mW cm^{-2}	7.0 min	10 s @600 mW cm^{-2}

After storage in the dark in a 0.9% (v/v) saline solution at 37°C for 1 wk, the restored teeth were simultaneously loaded with repeated thermal and mechanical stresses in a chewing machine (22). Thermal cycling was carried out in flushing water with temperatures changing 3,000 times from 5°C to 50°C, with a dwelling time of 2 min per temperature. The mechanical stress comprised 1,200,000 load cycles transferred to the center of the occlusal surface with a frequency of 1.7 Hz and a maximal load of 49 N applied by using a natural lingual cusp of an extracted human molar. Immediately after stressing, a second set of impressions was carried out of each restoration (President light body; Coltène-Whaledent). Subsequently, epoxy replicas were prepared for the computer-assisted quantitative margin analysis in a scanning electron microscope (XL20; Philips, Eidhoven, the Netherlands) at a magnification of ×200 to ×400 (22). The different marginal qualities were assessed as a percentage of the total length of margins analysed. The quality criteria 'continuous margin' and 'marginal gap' were mutually exclusive and amounted to 100%. The quality criterion 'marginal gap', which therefore is not separately graphed and listed, was further characterized, if appropriate, with the criteria 'marginal tooth fracture' and 'marginal restoration fracture', 'overhang' and 'underfilled margin'. Statistical analysis of the percentages of 'continuous margin' between groups was performed by using the Kruskal–Wallis test, with significance at a P-value of < 0.05. The differences between the percentages of 'continuous margin' before and after loading within a group were tested for significance by using the Wilcoxon Signed Rank test, with significance at a P-value of < 0.05.

Results

Table 2 summarizes the values of linear displacement induced by polymerization shrinkage and the polymerization force values, both measured 180 s after terminating the light polymerization procedure of each group. The difference between the lowest and highest polymerization force value was $\approx 20\%$. Group 1 (control), together with group 4, exhibited the highest shrinkage forces and also showed high linear displacement values. The linear displacement values ranged from 21.8 to 24.0 μm, being a difference of $\approx 12\%$.

Figures 1 and 2 represent the mean curves of the dynamic development of linear displacement and shrinkage force of each group. Figures 3 and 4 detail the
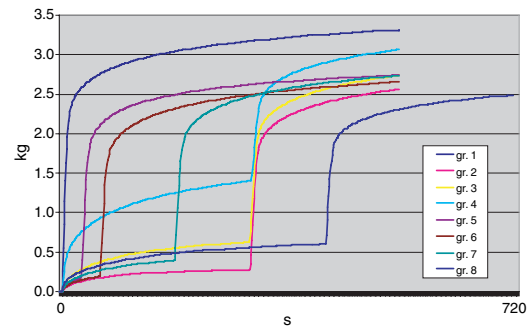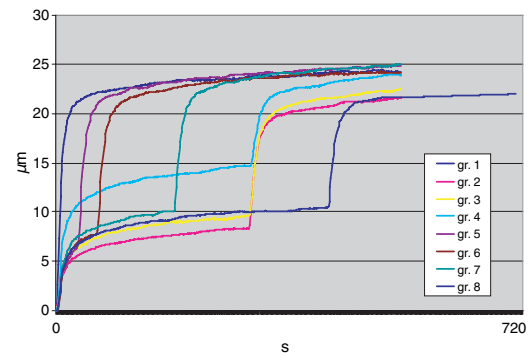

Fig. 1. Shrinkage force curves of the eight groups.

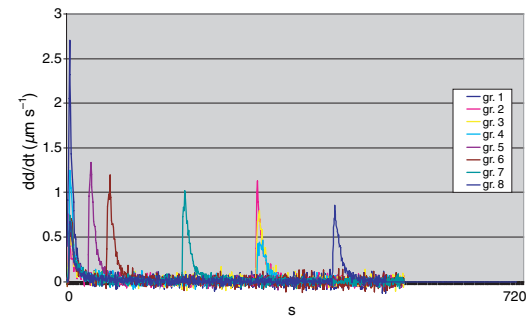*Fig. 2.* Linear displacement shrinkage curves of the eight groups.

Fig. 3. Linear displacement rate (first derivative of the linear displacement) of the eight groups.

Table 2

Shrinkage values of groups 1–8 at 180 s after the termination of light curing (n = 6)

Group	Linear displacement (μm)		Force (kg)	
	Mean ± SD	Different from groups*	Mean ± SD	Different from groups*
1 (control)	23.4 ± 0.5		3.0 ± 0.1	8, 6, 2, 5, 7
2	21.2 ± 0.5	5, 4, 7	2.5 ± 0.2	4, 1
3	22.1 ± 1.3		2.7 ± 0.3	
4	23.7 ± 1.6	2	3.0 ± 0.2	8, 6, 2, 5, 7
5	23.6 ± 1.0	2	2.5 ± 0.4	4, 1
6	23.2 ± 1.1		2.4 ± 0.2	4, 1
7	24.0 ± 1.7	2	2.6 ± 0.1	4, 1
8	21.8 ± 1.7		2.4 ± 0.1	4, 1

*Analysis of variance (ANOVA), Tukey-Kramer Multiple Comparison Test; P < 0.05.
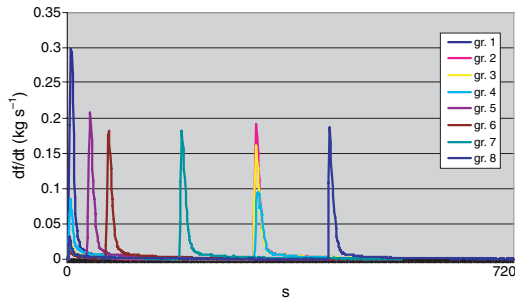SD, standard deviation

Fig. 4. Shrinkage force rate (first derivative of the shrinkage force) of the eight groups.

first derivative of the linear displacement and shrinkage force curves.

The percentages of 'continuous margin', before and after loading, are detailed in Table 3. Significant differences between groups were found for the enamel values after loading (Kruskal–Wallis, $P < 0.05$).

The influence of loading on percentages of 'continuous margin' was significant, except for enamel in Group 1 (Wilcoxon Signed Rank Test; $P < 0.05$).

The values for 'marginal tooth fracture', 'marginal restoration fracture', 'overhang' and 'underfilled margin' are not reported in detail because they were less than 5% in both experimental groups.

Discussion

Composites are subjected to postcuring for several hours after the termination of light polymerization (23). To standardize this effect for all groups of this study, a specific time-point was defined for the shrinkage force and linear displacement measurements, being always 180 s after termination of the light irradiation. This seems to be the latest point where postgel shrinkage rate reaches a constant level (24). According to the results, the shrinkage force and linear displacement were significantly dependent on the curing method applied. Thus, the first hypothesis of this study was confirmed, the results being in agreement with previous studies (14–16, 25). However, not all pulse-delay protocols applied in this study were significantly different from the control group, and the pulse-delay light curing protocols influenced linear displacement during shrinkage and shrinkage force in different ways. An explanation for this observation may be the fact that pulse-delay curing seems to modify the internal structure of the polymer network, making it more or less cross-linked, as per the specific parameters applied (26). The possible differences in the amount of cross-linking might have influenced shrinkage force and linear displacement differently (27). More in-depth studies may be needed to further clarify this phenomenon and to find an explanation for these differences.

The duration of the initial light curing and of the relaxation phase are thus additional influencing parameters of the pulse-delay method, besides the well-documented influence of power density (23, 28). In analogy to soft-start polymerization (29), the choice of the composite material seems also to play an important role in pulse-delay light curing (15). This is why the results of the present investigation may be limited to the specific composite and power densities used. Adding the fact that the difference of 1 s in the initial curing phase significantly influenced the shrinkage results, it might be extremely difficult for the practitioner to find and to apply the optimal combination of all mentioned parameters for his choice of composite material and light curing unit, especially in view of the fact that the literature is inconsistent on pulse-delay curing parameters. Under clinical conditions, the choice of a specific brand of composite with lower shrinkage (21) and a standard light curing protocol may therefore be more predictable than the simplistic use of the pulse-delay method.

It has been shown that pulse-delay curing reduces the shrinkage force rate of composites (16). The comparison between the control group (group 1) and all pulse-delay groups (groups 2–8) clearly confirmed this observation (Fig. 4). However, the amount of reduction in shrinkage force rate was not related to the absolute shrinkage force value. While the highest reduction in shrinkage force rate was found in group 4, this group exhibited the highest shrinkage force, not being significantly different from that of the control group. By comparing the shrinkage force rates of groups 2–4, it seems that the sum of the pulse and the final polymerization shrinkage force rates was almost equal, and that the values of these two rates depended on the duration of the pulse period. By extending the pulse period, the initial shrinkage force rate increased and the shrinkage force rate at the final polymerization decreased. As the two shrinkage force rates were almost equally as high in group 4, their maximum was the lowest. On the other hand, the shrinkage force increased by extending the pulse period, and it can be speculated that 3 s of pulse duration was already enough to sufficiently rigidify the composite, to reach similar stress values as the control.

Table 3

Percentages of 'continuous margin' of group 1 (control) and group 8 (pulse-delay) before and after thermo-mechanical loading (n = 8)

	Enamel		Dentin		Total	
	Before	After*	Before	After	Before	After
Group 1	89.2 ± 9.2	87.9 ± 12.2	92.8 ± 8.6	74.3 ± 21.0	91.0 ± 7.9	81.7 ± 15.9
Group 8	90.1 ± 5.9	68.8 ± 15.9	90.2 ± 10.3	60.2 ± 37.4	90.1 ± 6.5	62.8 ± 20.2

*Significant difference between the two groups (Kruskal–Wallis, $P < 0.05$)

Based on the results of the linear displacement and shrinkage force generated in the first part of this investigation, group 8 was chosen as the test group for the evaluation of marginal adaptation. The choice was based on the fact that this group showed the best combination of both low shrinkage force and low linear displacement during polymerization that were both significantly different from that of the control group (group 1). Even if group 2 exhibited lower linear displacement, it was not considered, for three reasons: the shrinkage force was higher than that in group 8; the linear displacement was not significantly different from that in group 8; and the composite after 1 s of initial curing was still slightly soft, so that in a clinical situation this procedure could not be used.

The second hypothesis of this study was disproved because no significant difference in marginal adaptation for the total marginal length of mixed class V restorations was observed, either before or after loading. This is despite the fact that the values of shrinkage force and linear shrinkage were significantly different and a reduction of almost 40% for shrinkage force rate and of almost 70% for linear displacement rate could be calculated in favor of group 8 in respect to group 1. One explanation may be the fact that the differences between the two groups were only 11.7% for linear displacement and 20% for force. As a result of the good reproducibility of the measuring methods, these values were significantly different; however, in absolute terms the differences were not very high. Another explanation could be the observation of a previous study where no significant differences were seen for different light curing procedures if using an incremental restorative technique in a class V cavity in contrast to bulk polymerization, where the differences were significant (30). Finally, light-curing modes may have little effect on the marginal seal of cervical cavities with a low C-factor (31), even if no significant difference between pulse-delay and standard curing was also found in class II cavities having a high C-factor, both before and after thermocycling (32). SAHAFI *et al.* showed a positive effect of pulse-delay curing on wall-to-wall contraction under light curing conditions similar to those used in some of the groups of the present investigation (17). However, SAHAFI *et al.* used cylindrical class V cavities purely in dentin, one single composite layer, different restorative/adhesive materials, no occlusal/thermal loading, and applied a different method of evaluation. Other studies also demonstrated a positive effect of the pulse-delay light curing on marginal adaptation (18, 20, 33). Again, different cavity configurations, curing conditions and restorative/adhesive materials were used, and no thermo-mechanical loading was applied.

The lower percentage of excellent margins after loading of the control group in comparison to a previous study (34) could be explained by the fact that the composite in this investigation was polymerized for 10 s (control) and 12 s (pulse-delay) per layer and therefore might have had a lower conversion rate (19). This occurred, at least in the second, occlusal layer, because the cervical layer was polymerized in total for 2×10 s in the control and for 2×12 s in the pulse-delay group, respectively, Despite this, the composite conversion rate can be assumed as having been similar in the control and in the pulse-delay group (19). Thus, this factor could not explain the larger decrease in marginal integrity owing to loading in the pulse-delay group compared with that of the control group. It is speculated that the reason for the inferior load resistance may lie in a less cross-linked polymer structure induced by the pulse-delay method, leading to different biomechanical properties of the composite (26, 27). This speculation merits further investigation in future studies.

In conclusion, pulse-delay light curing can significantly reduce both shrinkage force and linear composite displacement during polymerization. This effect depends on the exact times of the pulse and the relaxation period. However, even the best pulse-delay procedure of this study, in terms of shrinkage reduction, was not able to significantly improve marginal adaptation of mixed class V restorations, either before or after loading. In fact, a more pronounced tendency for disintegration of marginal adaptation after thermo-mechanical loading was observed in the pulse-delay group compared with the control, leading to a significantly lower marginal adaptation in enamel. Further research is needed to understand, in greater detail, the influence of different parameters on pulse-delay curing and to clarify their impact on marginal adaptation in different cavity classes, with different composite materials and under different restorative and loading conditions.

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