Scientific Research

Effect of Temperatures on Polymerization Stress and Microleakage of Class V Composite Restorations

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ABSTRACT

The loss of interfacial integrity was identified as one of the major causes for replacement of resin composite restorations. Preheating procedure has been proven to enhance flowability and adaptation of resin composites and increase their degree of conversion. The purpose of this study was to investigate polymerization contraction stress produced in resin composites after preheating to 37°C and 60°C, and measure microleakage of Class V restorations restored with preheated composites. Three resin composites (GC Kalore, Gradia Direct X, Filtek Supreme XT) at room temperature, 37°C, and 60°C were investigated. Maximum contraction stress of the composites ($n = 5$) was evaluated in a modified low-compliance device. Samples were light-cured for 40 seconds and the maximum force was recorded during 15 minutes. Calculations were done to adjust for the system's compliance and obtain linear shrinkage values of composites. Data were analyzed by Multivariated Analysis of Variance (MANOVA) and Tukey's test for multiple comparisons ($α = 0.05$). Seventy-two Class V cavities were prepared on the buccal surfaces of extracted premolars and divided into 9 groups. The teeth were restored with composites at 3 temperatures and were thermo-cycled between 5°C and 55°C with a one-minute dwell-time for 1000 cycles. The teeth were sealed with wax and nail vanish before placed in 0.5% toluidine blue dye for 24 hours. The teeth were embedded in self-curing resin and sectioned bucco-lingually with a slow-speed diamond saw, providing 3 sections per restoration. Microleakage was rated by two evaluators using a 0 - 4 ordinal scale at the occlusal and cervical margins under light microscope. Microleakage data were analyzed with Kruskal-Wallis ANOVA and Mann-Whitney U test ($α = 0.05$). Results indicate that preheating composites to 37°C and 60°C significantly increased polymerization contraction stress of composites ($p < 0.05$). A significantly greater amount of leakage was found at the cervical margins ($p < 0.05$). For all tested materials, preheating composites to 60°C resulted in significantly less microleakage at the cervical margin.

1. Introduction

Improvements in resin composites' mechanical properties and their reduced polymerization shrinkage during the past decade encouraged clinicians to use resin composites more frequently for posterior restorations. Majorities of improvements aimed to improve microstructure of the material including monomer composition, size, shape, and distribution of inorganic filler particles and targeted mainly at increasing the filler load of resin composites. However, increasing the filler load resulted in higher viscosity and led to concerns about handling, packing, and adaptation of the material. Many attempts were made to enhance composite adaptation and decrease microleakage between composites and dental cavity, either by using the flowable composite as a base material, chemical and laser treatments of dentin or by preheating the composites to lower their viscosity.

The effect of lower viscosity in improvements of adaptation has been proven important. This is the primary basis for producing flowable resin composites in which the lower viscosity can be achieved by decreasing their filler contents and making changes in the matrix chemistry, which severely reduce their physical properties. Many polymers exhibit lower viscosity when they are heated because thermal vibrations force the composite monomer further apart and allow them to slide by each other more

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readily. This property leads to a concept of warming or preheating composite resins before photopolymerization, which will decrease the viscosity and increase flow of resin composites. In addition, composites cured at elevated temperatures have been proven to increase polymerization rate and have a higher degree of conversion [1-3], which could result in improved mechanical properties [4]. Also, it has been reported that increasing composite temperature up to 60˚C might enhance the conversion degree on the top and in 2 mm of the bottom surfaces [2].

However, it has been reported that the increased degree of conversion associated with preheating would result in increased polymerization shrinkage [5]. One study has shown that temperature has a significant effect on polymerization shrinkage of microfilled composites. Preheating composites to relatively high temperatures (54˚C or 68˚C) causes a significant increase in volumetric shrinkage, but preheating composites to a body temperature causes similar shrinkage to that at room temperature [6]. Advantages of preheating the resin are to make more durable, highly filled, highly viscous conventional composite resin, to reduce the viscosity, to provide flow values that are similar to those of less filled, flowable resin composites, without undermining the mechanical properties. However, more investigation is necessary to determine the side effects of preheating procedures such as an increase of the polymerization shrinkage which causes stress at the tooth/restoration interface and may cause microleakage of the restorations. The main objective of this study was to evaluate the effect of preheating on polymerization contraction stress of composites and the *in vitro* marginal microleakage. It was hypothesized that increasing the preheating temperature would increase the polymerization stress and increase the microleakage at resin composites/tooth interfaces.

2. Materials and Methods

2.1. Polymerization Contraction Stress Measurement

The materials used in this study are indicated in **Table 1**. Polymerization contraction stress was measured by a modified low-complicance device (**Figure 1**), consisting of 2 parts; the first part is a load cell, which a brass steel piston was attached. A brass steel piston (10 mm diameter; 30 mm length) was used as the bonding substrate for the composite. The piston had one surface abraded with #180-grit sandpaper, coated with silane coupling agent prior to the application of a thin layer of unfilled resin (Adper™ Single Bond 2), and light-cured for 20 seconds (MiniLED, Satelec, France). A ringshape teflon mold was inserted at the end of the brass piston, created a cylindrical cavity (dimension 10 mm diameter × 1 mm

Table 1. Materials used in the present study, their respective batch number and manufacturer.

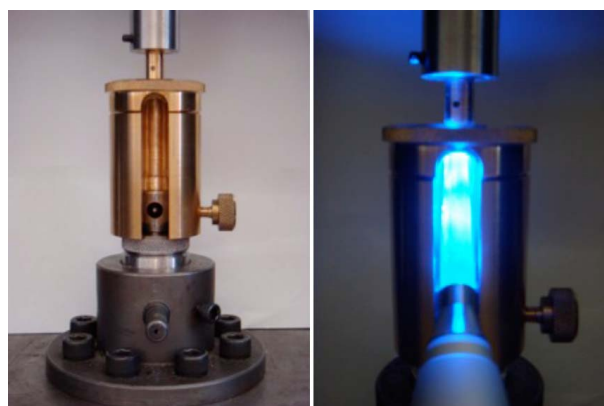| Material	Batch number	Manufacturer
Filtek™ Supreme XT	N151598	3M ESPE, Dental Products, St. Paul MN
GC KALORE™	910071	GC Corporation, Tokyo, Japan
Gradia™ Direct X	1201271	GC Corporation, Tokyo, Japan
Adper™ Single Bond 2	N283944	3M ESPE, Dental Products, St. Paul MN
Scotchbond™ Etchant	N287300	3M ESPE, Dental Products, St. Paul MN

Figure 1. The test setup for the polymerization stress measurements.

thickness) for resin composite. Uncured resin composite was preheated and inserted in the mold, the mold was then removed. The brass piston with the composite was heated up to specific test temperature on a thermal controlled plate, a thermocouple was placed inside a hole in the brass piston at all time to measure the composite temperature.

The second part is a brass cylinder, holding a clear perspex disc (22 mm diameter; 1 mm thickness). The brass cylinder has a slot which allowed for the placement of the light guide of a curing unit in contact with the perspex disc. The top surface of perspex disc was abraded with #180-grit sandpaper, cleaned, coated with a thin layer of unfilled resin (Adper™ Single Bond 2), and light-cured for 20 sec. When the desire temperature was reached, the brass piston was fixed to the load cell. The brass piston was driven down to just touched the perspex surface. The composite was photo-activated through the clear perspex disc. As the composite polymerized, contraction force was followed for 15 min. The force values were converted to nominal stress by dividing them by the cross-sectional area of the specimen (78.5 mm^2). Maximum contraction stress (Smax) was subjected to statistical analysis.

Data were analyzed by Multivariated Analysis of Variance (MANOVA), entering resin composite and temperature as main factors, and Tukey's test for multiple comparisons ($\alpha = 0.05$).

2.2. Microleakage Test

2.2.1. Tooth Selection
Seventy-two extracted caries and restoration-free permanent human premolars were selected, remaining soft tissue removed, and stored in deionized water for a maximum duration of 4 weeks. The teeth were cleaned with slurry of pumice and water, rinsed thoroughly with tap water, and then examined macroscopically with magnification for defects in the enamel and dentin.

2.2.2. Sample Preparation
The teeth were randomly assigned into nine groups of eight. A Class V preparation was made in the buccal surface of each tooth. The occlusal margin of the cavities were in enamel and the gingival margins located 1.5 mm apical to the cemento-enamel junction. Preparations were made with a 329 carbide bur in a high-speed handpiece equipped with water spray. Cavity dimensions were standardized (5.0 mm in width, 3.0 mm in height, and 2 mm in depth). For all groups, enamel and dentin are etched with 35% phosphoric acid gel (Scotchbond™ etchant, 3M Dental Products, MN, USA) for 20 s and 15 s respectively, rinsed for 20 s, and air-dried to obtain chalky-white appearance enamel and moist dentin. All cavities are treated with a resin-based adhesive system. The adhesive used in this study is Adper™ Single Bond 2 (3M ESPE, St. Paul, MN, USA). Wet-bonding technique is followed as recommended by the manufacturer, moist dentin was clinically evidenced by a uniform shiny surface on which water was not pooled. A fully saturated brush tip for each coat is used, applying two consecutive coats of Adper™ Single Bond 2 adhesive to prepared enamel and dentin. Later, the surface was dried gently for 5s and light cured for 20 s (miniLED, Satelec, France).

The cavities were restored as follow:

Group 1: restored with a room temperature Filtek Z350 XT;

Group 2: restored with a preheated (37˚C) Filtek Z350 XT;

Group 3: restored with a preheated (60˚C) Filtek Z350 XT;

Group 4: restored with a room temperature GC Kalore;

Group 5: restored with a preheated (37˚C) GC Kalore;

Group 6: restored with a preheated (60˚C) GC Kalore;

Group 7: restored with a room temperature Gradia direct X;

Group 8: restored with a preheated (37˚C) Gradia direct X;

Group 9: restored with a preheated (60˚C) Gradia direct X.

All restorations were done two increments with the first against the gingival wall, and light-cured for 20 s. Excess materials are removed with a No.170 bur, followed by finishing and polishing with the Softlex disk system (3M Dental Products Division, St. Paul, MN, USA). The restored teeth are stored in deionized water at 37˚C for 1 day before further treatment.

All samples are thermocycled for 1000 cycles between 5 and 55˚C with a dwell time of 1 min, before immersion in dye. The apices of the teeth are sealed with blue wax and coated with a nail polish 1 mm short of the restoration margins in order to reduce other leakage elsewhere that could lead to false positive results. The teeth were immersed in 0.5% toluidine-blue solution for 24 hour at room temperature. The superficial dye is removed with a pumice slurry and rubber cup after removal of the specimens from the dye solution. Teeth are then mounted in a cold-cure epoxy resin (Leco®, Leco Corporation, MI, USA) to facilitate handling during sectioning.

2.2.3. Microleakage Test
To measure the extent of microleakage, the teeth were sectioned longitudinally through the restorations in a bucco-lingual direction with a low speed diamond saw (IsoMet™, Buehler Ltd., Lake Bluff, IL, USA), providing 3 sections per restoration. The sectioned teeth were evaluated with a stereomicroscope (Leica MZ8, Leica Microscopy System Ltd., Heerbrugg, Switzerland) at 20 × magnification. The degree of microleakage determined through dye penetration was scored according to standardized criteria (0 to 4; **Table 2**, **Figure 2**). Double blinded evaluators measured the slices and then the Kappa test was performed. Differences in the frequency distribution of scores between groups were assessed using the Kruskal-Wallis test and assessments within the groups were assessed using the Mann-Whitney U test. The results of testing were analyzed with statistical software (IBM® SPSS® Statistics Base 21, IBM, USA). Significance is considered at the 0.05 level.

3. Results

Average polymerization contraction stress of composites at room temperature and when preheated to 37˚C and 60˚C are shown in **Table 3** and **Figure 3**. **Figure 4** shows trend lines of the polymerization contraction force during 0 - 60 second. Results indicate that preheating composites to 37˚C and 60˚C significantly increased polymerization contraction stress and the developmental rate of polymerization contraction force ($p < 0.05$). Statistically greater amount of leakage was found at the cervical margins compared to the occlusal margins ($p < 0.05$) for the group restored with composites at room temperature and the group restored with composites at 37˚C. No

Table 2. Microleakage scoring criteria.

0	No dye penetration
1	Dye penetration up to one-half of the cavity wall
2	Dye penetration up to total cavity wall
3	Dye penetration up to one-half of the axial wall
4	Dye penetration more than one-half of the axial wall

Table 3. Average polymerization contraction stress of composites at room temperature and when preheated to 37°C and 60°C.

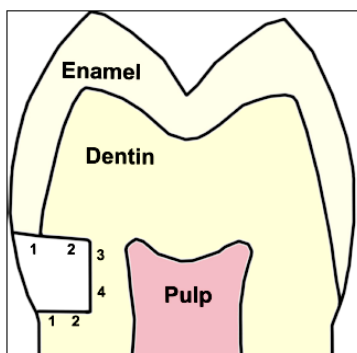| Materials	Room Temp	37°C	60°C
GC Kalore	4.18 ± 0.02	8.10 ± 0.07	9.85 ± 0.04
Gradia Direct X	5.61 ± 0.02	8.62 ± 0.03	10.72 ± 0.05
Filtek Supreme XT	11.83 ± 0.01	14.21 ± 0.05	16.39 ± 0.05

Figure 2. Diagram of Class V cavity and microleakage scoring.

statistical significant differences were observed among materials and temperatures at the occlusal margin. In contrast, for all materials tested, the 60°C preheated samples showed statistically lower microleakage at the cervical margin. In fact, there was no microleakage observed at the cervical margin for the group restored with composite preheated to 60°C.

The polymerization contraction stress results were analyzed using General Linear Model, Multivariated Analysis of Variance (MANOVA), Tukey's and Scheffe's Post Hoc test. Highly significant differences were found both between materials and between temperatures ($p < 0.01$).

The frequency distribution of different degrees of microleakage in the groups is shown in **Table 4**. There were significant differences between the microleakage scores for the enamel and dentin ($p < 0.05$). Less microleakage was observed at the occlusal margins than at the cervical margins. Only 2 restorations showed microleakage at the occlusal margins. There were no significant differences between materials and temperatures at the occlusal margins ($p > 0.05$). However, at the cervical margins, there
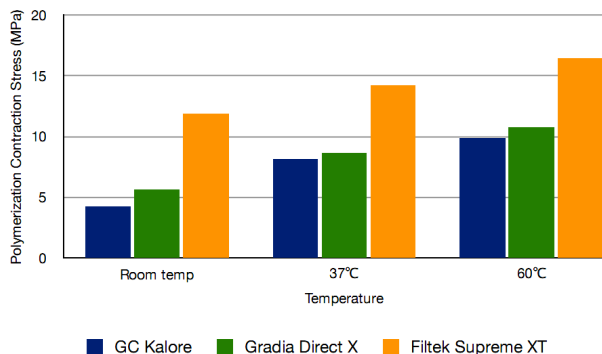

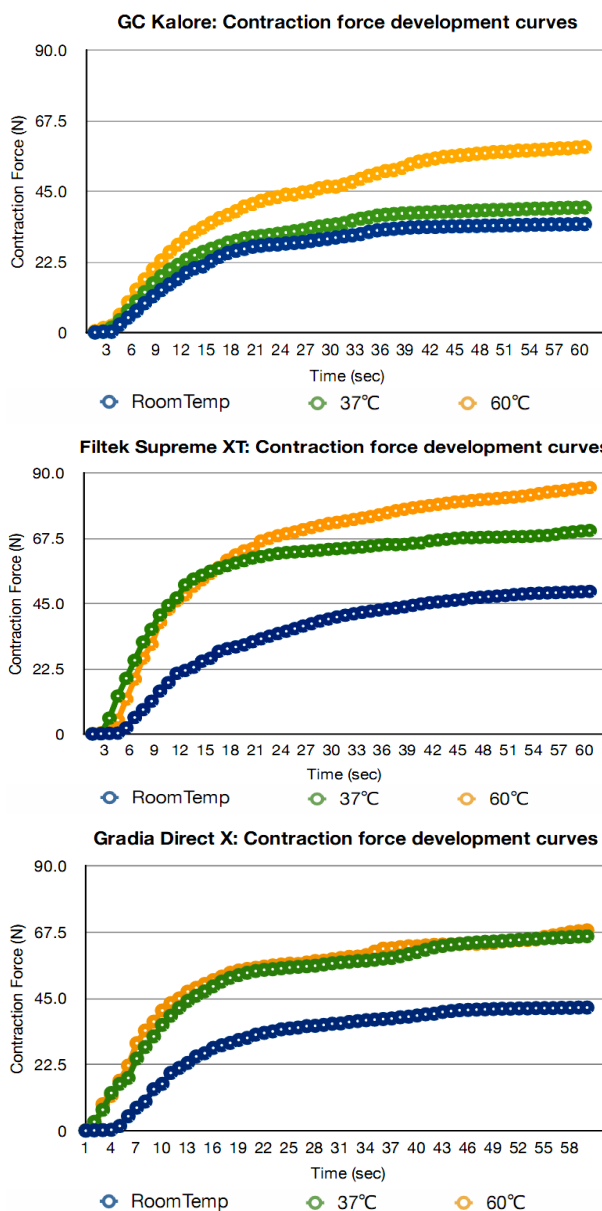**Figure 3. Polymerization contraction stress (MPa) of composites at different temperatures.**

Figure 4. Polymerization contraction force during 0 - 60 second.

Table 4. Frequency distribution of microleakage for each experimental group.

Group	Occlusal					Gingival				
	0	1	2	3	4	0	1	2	3	4
Group 1: Filtek Supreme (control)	7	1	0	0	0	4	3	0	1	0
Group 2: Filtek Supreme (37°C)	8	0	0	0	0	5	3	0	0	0
Group 3: Filtek Supreme (60°C)	8	0	0	0	0	8	0	0	0	0
Group 4: GC Kalore (control)	8	0	0	0	0	5	2	0	1	0
Group 2: GC Kalore (37°C)	8	0	0	0	0	6	2	0	0	0
Group 3: GC Kalore (60°C)	8	0	0	0	0	8	0	0	0	0
Group 5: Gradia Direct X (control)	7	1	0	0	0	5	3	0	0	0
Group 6: Gradia Direct X (37°C)	8	0	0	0	0	6	1	0	0	1
Group 7: Gradia Direct X (60°C)	8	0	0	0	0	8	0	0	0	0

were significant differences among the preheated groups ($p < 0.05$). Preheat composites to 60°C significantly reduced the degree of cervical leakage in all materials tested. In fact, no microleakage was observed at the cervical margin in this group.

Figure 5 show representative stereomicroscopic images of the samples with and without cervical microleakage.

4. Discussion

The objective of this experimental study was to evaluate the effect of preheating temperatures on polymerization contraction stress of composites and to investigate the extent of *in vitro* marginal microleakage of Class V resin composite restoration restored with preheated composites as compare to the cavity restored with room temperature composites. The extent of leakage after thermal cycling is relevant to clinical practice since microleakage of saliva, oral fluids and bacteria at the tooth-restoration interface has been linked causally to a range of problems, including marginal staining, postoperative sensitivity, and secondary caries. The integrity and durability of the marginal seal is an important factor in the longevity of adhesive dental restorative materials, particularly for composite resins. The absence of a seal at restoration margins permits the entry of oral bacteria and fluids, which can result in postoperative sensitivity, adverse pulpal responses and recurrent caries [7]. In the present study, standardized methods were used to minimize confounding factors. The dye penetration test is the most widely used laboratory method for assessing leakage, and for the purposes of this study a simple grading system was used [8]. The apical extent of the test cavities was intentionally placed into the root surface because leakage at this site is known to be a clinical concern when Class II and Class V cavities are restored with composite resin materials.

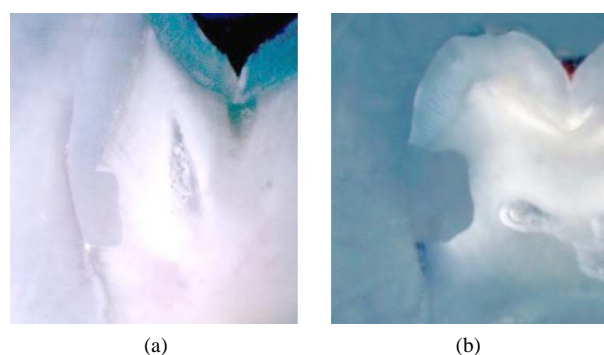The results show that preheating composites signifi-

(a)   (b)

Figure 5. Representative stereo-microscopic images of the cavity with microleakage (a) and without cervical microleakage (b).

cantly increased polymerization contraction stress of composites and for all materials tested, preheating to 60°C resulted in significant reduction of microleakage at the cervical margin. This is agreed with Fróes-Salgado *et al.* who evaluated the effect of pre-heating on marginal adaptation, monomer conversion, flexural strength, microhardness, and polymer cross-linking of a resin composite under a non-isothermal condition. They found that under non-isothermal conditions (similar to a clinical situation) preheating composite to 68°C did not improve the degree of conversion, flexural strength or polymer crosslinking, but yielded enhanced marginal adaptation [9]. Wagner *et al.* also found that preheating composites can improve adaptation of resin composites to tooth structure and significantly reduced microleakage, although delay of light curing after placement appears to be counterproductive and diminishes the positive effects from the preheating treatment [10]. They also concluded that flowable liner was less effective than preheating the composite in reducing microleakage. Interestingly, the previous expectation that composite preheating could worsen the marginal adaptation of composites to the cavity walls due to the increased conversion rates [1,2] and consequently increased polymerization shrinkage [11,12]

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was not confirmed by the results of the present investigation. The results shows that preheating composite to high temperatures lead to an increase in polymerization contraction stress but less microleakage at the cervical margin. This is in contrast with several studies that have shown a direct relationship between contraction stress and marginal leakage in resin composite restorations [13-15]. This was also expected because resin composites exhibit a six to eight times greater thermal expansion than the surrounding tooth structures [9,16], polymerization shrinkage along with thermal contraction might create high interfacial stresses in preheated composites upon thermal equilibrium, with detrimental effects on marginal adaptation, integrity and seal [13]. Further investigation is therefore needed regarding advantages and disadvantages of dental composite preheating before we can make a conclusion if this method is appropriate in dental practice.

5. Conclusion

The present finding suggests that in the challenging situation of the cervical restoration which extends onto the root surface, preheating composites to 60˚C significantly reduced microleakage at the tooth-restoration interfaces. Preheating however results in non-desirable increase of polymerization contraction stress. More investigation should be done regarding the consequence of the increased stress at the tooth restoration interfaces to the strength of the restored tooth.

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