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Post-gel Shrinkage Strain and Degree of Conversion of Preheated Resin Composite Cured Using Different Regimens

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Clinical Relevance

The preheating of resin composite prior to curing yielded a better degree of conversion but had a negative impact on the induced post-gel shrinkage strain; soft-start mode is recommended, as it reduces the post-gel shrinkage strain without affecting the degree of conversion.

SUMMARY

Objectives: This study investigated the influence of resin composite preheating temperature, light curing regimen (mode and duration) on post-gel shrinkage strain (PGSS) and degree of conversion (DC) of a restorative resin composite.

Methods: A hybrid resin composite preheated to three different temperatures (37°C, 54°C and 68°C) was cured using a high intensity light emitting diode (LED) curing unit. The light source was used in 40- and 20-second continuous mode as well as soft start mode. The resin composite was monitored for PGSS during curing and 10 minutes following light irradiation using strain gauges. DC was measured using FTIR spectrometer. The results were analyzed using two-way ANOVA followed by the Duncan's Multiple Range Test for pairwise comparisons. The significance level was set at $p \leq 0.05$.

Results: Preheating of the resin composite significantly increased its PGSS and DC. The soft start groups revealed significantly lower PGSS values compared to the 20- and 40-second groups without altering the DC.

Conclusions: Preheating of resin composite prior to curing increased its DC but also increased its PGSS. The soft-start mode decreased the PGSS of resin composite without altering the DC.

INTRODUCTION

One of the recent innovations in resin composite application involves preheating of resin composite material before insertion into the cavity.[1] Preheating of resin

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composites before use with a chairside thermal assist unit shows an improvement in composite properties and reduces curing time.[2-3] It also makes them easier to place and adapt to the walls of the prepared cavity due to increased flow.[4-5] One added benefit of resin composite preheating is the enhancement of the maximal polymerization rate and the overall monomer conversion.[6] Issues regarding the use of composite preheating need to be investigated so that the clinician can better understand the variables associated with this method.[7]

A quartz tungsten halogen (QTH) light source was the predominant type of light for polymerization of resin composite restorative materials.[8] Despite its popularity, halogen bulb technology has several shortcomings, including limited effective lifetime, production of large quantities of heat during polymerization cycles that results in degradation of the bulbs, reflectors and filters.[9-13] To overcome such problems, LED technology has been proposed,[10-12] offering advantages, including long life expectancy with reliable light intensity, less heat generation and good resistance to shock and vibration.[14] Technical advancements in LED curing units have been rapid in the past few years. The first generation units consisted of an array of relatively low-powered chips offering a comparatively low output and poor curing performance compared with conventional QTH lights.[15-16] However, second generation LED models demonstrate better performance, using a single chip with a significantly higher surface area that emits only one color range of vastly increased output power.[12,17]

All contemporary resin composite restorative materials shrink during polymerization, resulting in a volumetric reduction ranging from 1.5% to 5%, depending on the molecular structure of the monomer, the amount of filler, rate of cure[18] and degree of conversion of the resin matrix.[19] The total shrinkage can be divided into two components: the pre-gel and post-gel phases. During pre-gel polymerization, the composite is able to flow, which relieves stresses within the structure.[20] After gelation, flow ceases and cannot compensate for shrinkage stresses. Post-gel polymerization, therefore, results in clinically significant stresses in resin composite-tooth bond and the surrounding tooth structure.[21]

Several curing protocols have been suggested to reduce resin composite shrinkage stress that can disrupt the bond to the cavity wall. The so-called soft start polymerization characterized by using an initial low-power intensity of the curing light followed by higher-power intensity has been advocated to minimize internal stresses in composite and improve its marginal adaptation.[22-23]

Hence, the current study has been carried out to assess the post-gel shrinkage strain (PGSS) and degree of conversion (DC) of a resin composite preheated to three different temperatures (37°C, 54°C and 68°C) and cured with a LED curing unit using three different curing regimens: (40 and 20 seconds) continuous mode and soft start mode.

METHODS AND MATERIALS

Measurements of Post-gel Shrinkage Strain

A hybrid resin-based composite restorative material (Tetric Ceram HB, Vivadent, Schaan, Liechtenstein, Lot #J22359 of shade-A3) was used. The test setup (Figure 1) included a white Teflon frame with a 7 mm inner length, 4 mm inner width and 2 mm height that was used to circumscribe the resin composite specimens. The Teflon frame was chosen so as not to adhere to the resin composite, thus allowing its free shrinkage. A glass slide served as a base for the setup. A foil electrical resistance strain gauge (Strain Gauges, Kyowa Electronic Instruments Co, LTD, Tokyo, Japan, Lot #Y4003S) was placed onto the flat glass surface.[24-26] The gauge was 2 mm in length and had an electric resistance of 120W and a gauge factor of 2.09 ± 1.0%.

Resin composite restorative material was placed in the cavity of the Teflon frame each time, with the strain gauge centralized in place. Care was taken to ensure complete filling of the frame followed by placement of a Mylar polyester strip (Hawe-Neos Dental, Bioggio, Switzerland), then the excess composite material was extruded using pressure applied through a second glass slide that was then removed. The foil strain gauge was connected to a strain-monitoring device (Strain-Meter PCD-300A Kyowa-Electronic Instruments Co, LTD, Tokyo, Japan) initially balanced at zero.

Sixty specimens were prepared and grouped into three groups of 20 specimens each. Based on the curing regimen (mode and duration) used, the specimens were grouped into continuous mode for 40 seconds, continuous mode for 20 seconds and soft start mode (10 seconds at 400 mW/cm^2, followed by 30 seconds 1400 mW/cm^2). Each group was further subdivided into four subgroups of five specimens each, according to the resin composite preheating temperature, including resin composite applied at room temperature (23 ± 1°C) (control) and resin composite preheated to temperatures 37°C, 54°C and 68°C prior to application. Preheating of the resin composite was accomplished using a chairside pre-

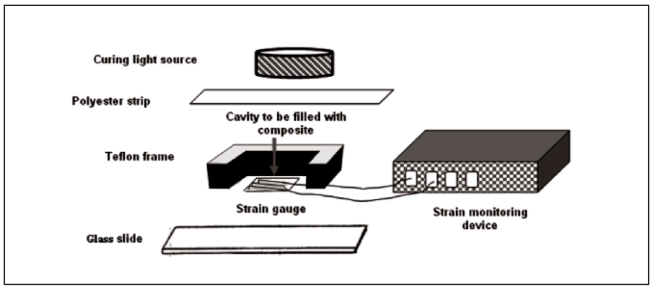

Figure 1: *The test setup used for PGSS measurement.*

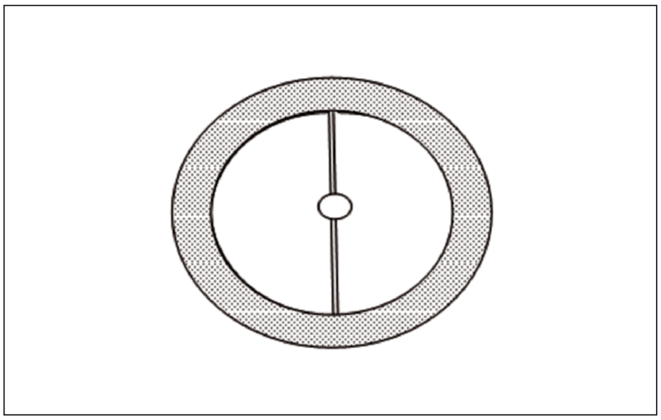

Figure 2: *The mold used for preparation of the DC specimens.*

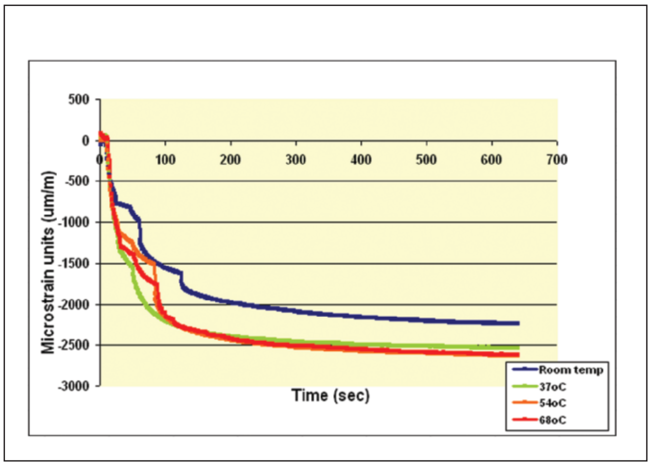

Figure 3: *Strain versus time curves for resin composite preheated to different temperatures cured for 40 seconds.*

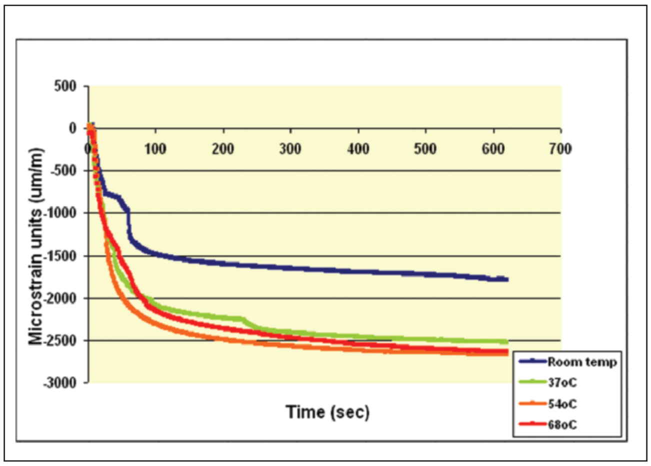

Figure 4: *Strain versus time curves for resin composite preheated to different temperatures cured for 20 seconds.*

heating device (Calset Thermal Assist Unit, Ad Dent Inc, Danbury, CT, USA). The resin composite was cured using LED curing unit (ARTESYN, ARTESYN Technologies APZS, LR41062, China) with an intensity of 1400 mw/cm^2. The intensity of the light-curing source was checked using LED radiometer (Kerr Corporation, Orange, CA, USA).

Strain measurements for each experimental condition (n=5) were recorded during curing and 10 minutes following light irradiation. Strain versus time curves for the different testing conditions were obtained using strain meter software—PCD30-A (Kyowa-Electronic Instruments Co, LTD, Tokyo, Japan).

Spectroscopic Measurement of the Degree of Conversion

DC was measured using a Fourier Transform Infra-Red (FTIR) spectrometer (Thermo-Nicolet Nexus 670 FTIR, GMI, Inc, Ramsey, MN, USA). A total of 60 specimens were prepared and grouped as described previously. The specimens were prepared by polymerizing the material in a disc-shaped split white Teflon mold 2 mm thick and 4 mm in diameter (Figure 2) and cured through Mylar polyester strips according to the curing regimens described in the previous section.

Five disc specimens were prepared for each experimental condition (n=5) and stored dry at room temperature in a light-proof container for 24 hours before testing. Each specimen had 32 scans at a resolution of 4 cm^{-1} and gave a blot of wave number from 4000-400 cm^{-1} against absorbance peak intensities using OMNIC 5.1 software on a computer connected to the FTIR unit. The DC for each experimental condition was determined by calculating the peak intensities of aliphatic carbon double bonds (C=C) at 1636 cm^{-1} and aromatic C=C at 1609 cm^{-1} for unpolymerized material and those after polymerization. The peak intensities as ratios of the uncured and cured material were calculated, and DC was expressed in a percentage relative to the uncured state. The DC percentage was calculated according to the equation given by Plian and others.[27]

$$\%\ DC = 1- \frac{(\text{aliphatic C=C/aromatic C=C}) \text{ of polymer x } 100}{(\text{aliphatic C=C/aromatic C=C}) \text{ of monomer}}$$

Statistical Analysis

The Statistical Package for Social Sciences (SPSS) version 12.0 was used for statistical analysis of the data. Two-way ANOVA was used to compare the mean PGSS values and the mean percentage DC values for the resin composite. When the results of ANOVA were significant, the Duncan's Multiple Range Test was used for pairwise comparisons. The significance level was set at $p \leq 0.05$.

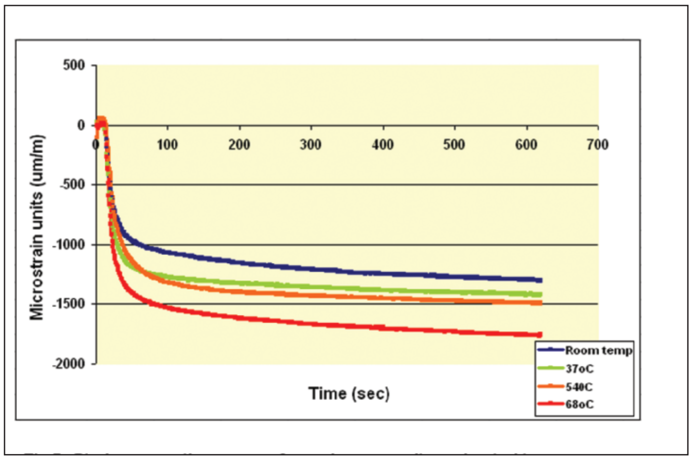

Figure 5: *Strain versus time curves for resin composite preheated to different temperatures cured using soft start mode.*

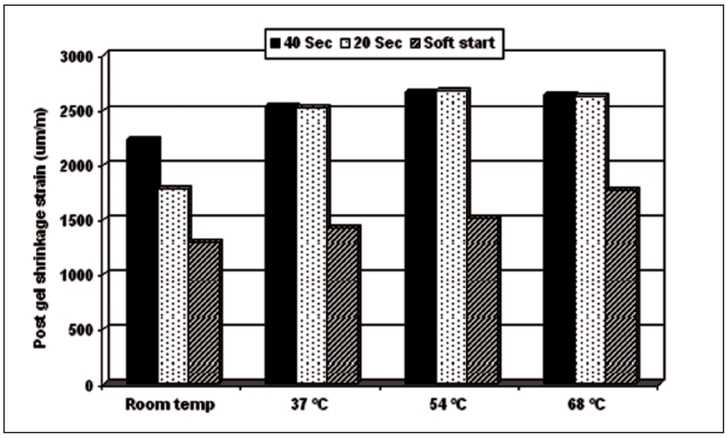

Figure 6: *Mean post gel shrinkage strain (μm/m) for different regimens within each preheating temperature.*

RESULTS

Results of Post Gel Shrinkage Strain

Strain versus time curves for different resin composite preheating temperatures with different curing regimens are shown in Figures 3-5. The curves demonstrated tensile strain (expansion) in the early phases of polymerization that became compressive (contraction) as polymerization progressed. A sharp increase in compressive strain was noted during and directly after turning off the light source, followed by a gradual increase, ending with a plateau.

Two-way ANOVA showed a highly significant effect for resin composite preheating temperature, as well as light curing regimen as independent variables on PGSS of resin composite ($p \leq 0.001$). Also, the interaction between the two variables was significant. Means, standard deviations and test of significance for the effect of curing regimen and preheating temperature on PGSS (μm/m) are shown in Table 1. Comparing the effect of resin composite preheating temperature within each curing regimen revealed a significant increase in PGSS for the 37°C, 54°C and 68°C groups compared to the room temperature group for all curing regimens, where the 54°C and 68°C groups were significantly higher than 37°C. On the other hand, comparing the different curing regimens (Figure 6) revealed significantly lower mean PGSS values for the soft start groups compared to the 20- and 40-second groups for the different preheating temperatures tested. The latter two groups showed significant differences at room temperature; however, the differences between them were insignificant at 37°C, 54°C and 68°C preheating temperatures. Ranking of the different experimental groups according to the effect of preheating temperature and curing regimen on their PGSS (μm/m) values is shown in Table 2. From Table 2, it can be deduced that the lowest mean PGSS values were obtained with the soft start mode regimen, where the lowest mean value was shown by the room temperature group followed by the preheated resin composite groups.

Results of the Degree of Conversion

Two-way ANOVA showed a highly significant effect of preheating temperature and light curing regimen as independent variables on the mean percentage DC of resin composite ($p < 0.001$). Means, standard deviations

Table 1: *Means, Standard Deviations (SD) and Test of Significance for the Effect of Curing Regimen and Preheating Temperature on Post-gel Shrinkage Strain (μm/m) of Resin Composite*

Temperature	**Room Temp**			**37°C**			**54°C**			**68°C**		
Regimen	**Means (SD)**	**D1**	**D2**	**Mean (SD)**	**D1**	**D2**	**Mean (SD)**	**D1**	**D2**	**Mean (SD)**	**D1**	**D2**
40 Seconds	-2230.4 (25.2)	c	a	-2541.5 (19.9)	b	a	-2660.0 (16.4)	a	a	-2638.1 (16.6)	a	a
20 Seconds	-1783.6 (30.6)	d	b	-2525.6 (33.7)	c	a	-2683.0 (30.0)	a	a	-2628.5 (40.8)	b	a
Soft start	-1298.9 (24.9)	d	c	-1427.0 (20.4)	c	b	-1509.4 (24.3)	b	b	-1769.0 (14.9)	a	b

D1= Duncan's Multiple Range Test for the effect of temperature.
Means with the same letter within each row are not significantly different.
D2= Duncan's Multiple Range Test for the effect of curing regimen.
Means with the same letter within each column are not significantly different.

Table 2: *Means, Standard Deviations (SD), Ranking and Test of Significance for the Effect of Preheating Temperature and Curing Regimen on Post-gel Shrinkage Strain (μm/m) of Resin Composite*

Experimental Condition	Means	SD	Maximum Strain	Minimum Strain	DT
54°C + 20 Seconds	-2683.0	30.0	-2718.2	-2638.9	A
54°C + 40 Seconds	-2660	16.4	-2675.0	-2633.8	A
68°C + 40 Seconds	-2638.1	16.6	-2662.7	-2619.4	B
68°C + 20 Seconds	-2628.5	40.8	-2685.6	-2579.8	B
37°C + 40 Seconds	-2541.5	19.9	-2563.5	-2510.0	C
37°C + 20 Seconds	-2525.6	33.7	-2568.7	-2486.2	C
Room temperature	-2230.4 + 40 Seconds	25.2	-2260.5	-2198.8	D
Room temperature	-1783.6 + 20 Seconds	30.6	-1825.2	-1746.1	E
68°C + Soft start	-1769.0	14.9	-1790.4	-1750.4	E
54°C + Soft start	-1509.4	24.3	-1538.5	-1480.8	F
37°C + Soft start	-1427.0	20.4	-1450.6	-1400.0	G
Room temperature + Soft start	-1298.9	24.9	-1328.5	-1268.2	H

DT= Duncan's Multiple Range Test for the effect of the experimental condition.
Means with the same letter within each column are not significantly different.

and test of significance for the effect of preheating temperature and light curing regimen on the mean percentage DC of resin composite are shown in Table 3. Comparing the effect of resin composite preheating temperature revealed a significant increase in mean percentage DC values for the 37°C, 54°C and 68°C groups compared to the room temperature group for all curing regimens. Statistical analysis revealed an insignificant difference between the 54°C and 68°C groups, however, both groups were significantly higher than the 37°C group. Concerning the different curing regimens (Figure 7), the 40-second groups were significantly higher than the 20 second-groups at room temperature, 37°C and 68°C; however, the soft start groups were not statistically different from either of the other regimens for any of the experimental conditions. For the 54°C groups, there were insignificant differences among the three curing regimens.

DISCUSSION

According to the current literature, the polymerization stress of resin composites is determined by their volumetric shrinkage, viscoelastic behavior and restrictions imposed to polymerization shrinkage. Therefore, the material's composition, its DC and reaction kinetics become aspects of interest.[19]

Temperature has shown to have a significant effect on the final conversion values of resin composites, where the results of the current study revealed a significant increase in the DC of resin composite by increasing its preheating temperature. This is in agreement with Lovell and others[28-29] and Daronch and others.[30,36] The increased conversion with temperature may be attributed to the decrease in system viscosity associated with increasing temperature, which enhances radical mobility, resulting in additional polymerization and higher conversion.[4,28-29] Furthermore, as composite temperature is raised, additional free volume increases, giving trapped radicals increased mobility, resulting in further conversion. However, plateaus (no further increase) were detected above 54°C, indicating that excessive preheating may not be beneficial.

Table 3: *Means (SD) and Test of Significance for the Effect of Preheating Temperature and Light-curing Regimen on Degree of Conversion (%)*

Temperature	Room Temp			37°C			54°C			68°C		
Regimen	**Means (SD)**	**D1**	**D2**	**Mean (SD)**	**D1**	**D2**	**Mean (SD)**	**D1**	**D2**	**Mean (SD)**	**D1**	**D2**
40 Seconds	72.86 (0.65)	c	a	75.03 (0.56)	b	a	78.18 (0.44)	a	a	78.28 (0.52)	a	a
20 Seconds	71.02 (1.39)	c	b	73.55 (0.60)	b	b	77.59 (0.55)	a	a	77.39 (0.65)	a	b
Soft start	72.34 (0.88)	c	ab	74.28 (0.91)	b	ab	77.67 (0.31)	a	a	77.76 (0.43)	a	ab

D1= Duncan's Multiple Range Test for the effect of temperature.
Means with the same letter within each row are not significantly different.
D2= Duncan's Multiple Range Test for the effect of regimen.
Means with the same letter within each column are not significantly different.

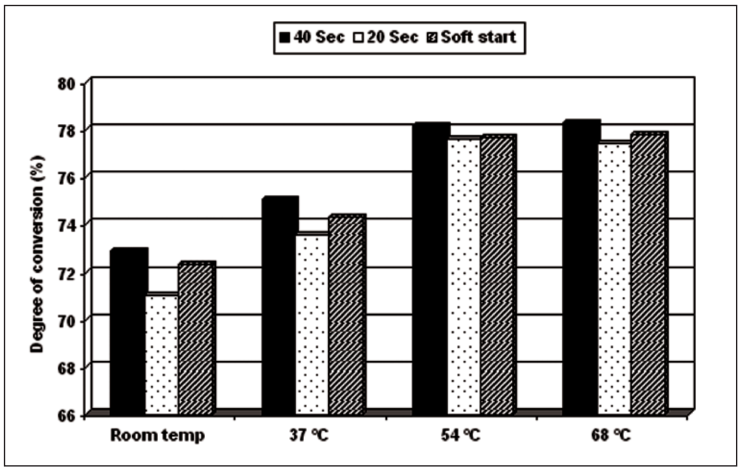

Figure 7: *Mean degree of conversion (%) for the different regimens within each preheating temperature.*

It is given that the polymerization of dimethacrylate resins results in shrinkage. Some shrinkage occurs during the predominantly viscous phase, prior to development of the elastic modulus of the resin, and some shrinkage occurs after the elastic behavior dominates. Total contraction refers to the measurement of shrinkage throughout the polymerization reaction from a viscous fluid state (pre-gel), through gelation to post-gel and finally, vitrification.[31]

Although shrinkage occurs in the pre-and post-gel phases, it is clinically significant only after measurable stiffness develops, when shrinkage forces can be transferred to the surrounding structures. The force transfer challenges the composite-tooth bond,[32-33] resulting in damage to the bond, microleakage, post-operative sensitivity and secondary caries.[34] If the restoration-tooth bond remains intact, stresses induced by resin composite shrinkage may cause deformation of the surrounding tooth structure.[35-37]

If the elastic modulus is developed early in the curing cycle, the gel point shifts in time so that the proportion of post-gel to pre-gel contraction increases. Although the total volumetric contraction, which includes pre- and post-gel contraction, may not change, the post-gel contraction of materials with an earlier onset of elastic modulus will be higher relative to materials with a slower onset. This emphasizes the importance of post-gel contraction, which provides an indication of stress induction into the surrounding tooth structure.[38] The recording of only post-gel contraction requires a sensor that ignores viscous flow and is sensitive only to shrinkage of a solid with measurable stiffness. The strain gauge is such a sensor.[31] These gauges are thought to measure shrinkage of the composite only after the composite develops elastic properties. Since the metallic foil of the strain gauge is mounted on a relatively stiff polyamide backing, the gauge measures dimensional changes only after the composite develops some stiffness, where the elimination of clinically less significant pre-gel shrinkage from the measurement can be viewed as an advantage of the strain gauge method.[20,39]

In the current study, strain versus time curves for resin composite cured with high intensity LED at different temperatures using different curing regimens revealed initial tensile strain (expansion) during the early phases of polymerization, which indicates thermal expansion of the restorative material.[38] This thermal expansion may be attributed to the effect of heat generated by the curing light source[40] in addition to the effect of resin composite preheating temperature, which causes further thermal expansion. This was followed by compressive strain (contraction) during light curing, when the resin rapidly sets and thus acquires rigidity due to chain lengthening and cross linking within the matrix. After turning off the light source, contraction continues, probably due to continuation of the polymerization reaction added to the loss of radiant heat as previously described by Chen and others,[41-42] as well as the effect of temperature decrease due to cooling of the resin composite to room temperature.

The significant increase in PGSS of all preheated resin composite groups compared to room temperature groups may be attributed to three concurrent factors. First, it is likely that there was a rapid stress buildup within the composite due to a faster rate of polymerization as a result of preheating and rapidly reaching the gel point.[43-45] Second, the higher DC values due to preheating lead to an increase in volumetric shrinkage and elastic modulus of the material.[46] Third, the effect of high thermal shrinkage of the heated composite as it cools to room temperature added to polymerization shrinkage may contribute to the dramatic increase in induced stresses. This explanation was confirmed by El-Hegazi,[47] who found that polymerization shrinkage increases as temperature increases. The rapid rate of polymerization may explain the insignificant differences in mean PGSS values between 40- and 20-second regimens for each of the preheated composite groups (37°C, 54°C, 68°C), which may indicate that the effect of preheating has surpassed influence of the difference in energy density.

A light-curing unit with high intensity is recommended almost universally. Generally, these recommendations are based on curing depths and physical properties of the resin composite. However, they do not consider the possible negative effect of high intensity lights on stress development.[48] The significant increase in PGSS and the increase in DC of resin composite may, in part, be attributed to the effect of the light energy of the curing light source, in addition to the previously mentioned reasons, where the higher energy produced a higher polymerization rate and greater final contraction. This agrees with Correr and others,[49] who reported that DC depends on the amount of energy supplied to the composite and that low energy density produces composites with low DC. This may further explain the significant decrease in DC of the 20-second cure groups compared to the 40-second groups.

Use of the soft-start polymerization regimen resulted in a significant decrease in PGSS compared to groups cured with a continuous high intensity irradiation regimen (40- and 20-second groups). This may be attributed to the fact that use of a soft-start polymerization regimen reduces the polymerization rate and extends the time allowed for viscous flow and, consequently, for non-rigid shrinkage.[43] However, this does not affect the DC.

The results of the current study revealed that strain buildup in resin composite was affected by its preheating temperature and its DC, which may, in turn, vary according to the curing technique. Nevertheless, further studies for the clinical significance of preheating resin composite and its curing with high intensity LED units are still required.

CONCLUSIONS

Under the conditions of this *in-vitro* study, the following could be concluded

1. Preheating of resin composite prior to application increased its DC but also increased its PGSS.
2. Soft start mode decreased the PGSS of resin composite without altering the DC.

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