

Effect of pre-heating on the viscosity and microhardness of a resin composite

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SUMMARY The effect of pre-heating resin composite on pre-cured viscosity and post-cured surface hardness was evaluated. Groups of uncured specimens were heated to 60 °C and compared with control groups (24 °C) with respect to viscosity and surface hardness. Mean (SD) viscosities of the pre-heated specimens ($n = 15$) were in the range of 285 (13)–377 (11) (Pa) compared with 642 (35)–800 (23) (Pa) at ambient temperature. There was a statistically significant difference between the two groups ($P < 0.001$). Mean (SD) Vickers microhardness (VHN) of the pre-heated group ($n = 15$) was 68.6 (2.3) for the top surface and 68.7 (1.8) for the bottom surface measured at 24 h post curing (specimen thickness = 1.5 mm). The corresponding values for the room temperature group

were 60.6 (1.4) and 59.0 (3.5). There was a statistically significant difference between corresponding measurements taken at the top and bottom for the pre-heated and room temperature groups ($P < 0.001$). There was no significant difference between top and bottom measurements within each group. Pre-heating resin composite reduces its pre-cured viscosity and enhances its subsequent surface hardness. These effects may translate as easier placement together with an increased degree of polymerization and depth-of-cure.

KEYWORDS: resin composite, surface hardness, viscosity, pre-heating, ease of placement, hardness ratio

Accepted for publication 1 December 2009

Introduction

Placement of resin composite (RC) restorations in anterior and, in particular, posterior teeth has increased dramatically over the past 5 years (1). This has been driven by factors such as patient demand, an increased desire for minimally invasive restorations and more predictable dental adhesive systems (2). Indeed, the longevity of load-bearing RC restorations placed in posterior teeth may be comparable to that of amalgam (3, 4). However, it is of concern that when using more viscous compositions, such as high-filler content densified or hybrid RC materials, these may not adapt fully or completely to the cavity preparation (5). In such scenarios, voids may be introduced between the completed RC restoration and underlying tooth surface, which may result in poor marginal integrity (6).

However, the viscosity of more viscous RC materials may be reduced through pre-heating, before placement and polymerization, to a temperature of approximately 68 °C (7). A decrease in viscosity between 20 and 35 °C can also occur (8). A serendipitous aspect of such pre-heating has been shown to be improved polymerization parameters such as increased reaction rate and degree of conversion (9–11). Thus, it might be expected that pre-heating, before polymerization, would improve the mechanical properties of RC materials and such an effect has been reported (12, 13). Additionally, a correlation has been reported between surface microhardness and degree of polymerization (14) such that the former (e.g. Vickers hardness number, VHN) may be appropriate as a simple monitor of mechanical strength.

Pre-heating may be achieved by placing compoules, or syringes, of the RC material in a composite warming

tray or a water bath. At least one manufacturer presents a dedicated composite heater for this purpose (15). Warming RC has also been shown to reduce the film thickness of some conventional materials (16), which serves to increase ease of manipulation and has resulted in less microleakage *in vitro* (17). Indeed, the kinetic parameters for polymerization of resin monomers have been shown to follow an Arrhenius-type behaviour such that a relatively modest increase in temperature may promote a large increase in reaction rate (18). Recent reports indicate an increase in microhardness for commercially available RC materials with pre-heating, but this is not accompanied by complementary viscosity data. (12, 19). A comparison of top and bottom surface hardness of disc specimens, where the activation curing tip is in contact with the top surface only, may be taken as an indication of the 'depth-of-cure' (19), and it is suggested that the bottom/top ratio should be in excess of 0.8 for adequate polymerization (20).

The purpose of this investigation was to compare

- 1 pre-cure viscosity
- 2 post-cure top and bottom surface microhardness numbers (VHN)
- 3 post-cure bottom surface/top surface VHN ratios of a commercial densified (high-filler content) RC material exposed and cured at a nominal temperature of 60 °C with those exposed and cured at room temperature.

The null hypotheses are that

- 1 there is no difference in mean pre-cure viscosity for a RC measured at 60 and 24 °C.
- 2 there is no difference in post-cure mean surface microhardness for discs of a RC (top and bottom surface, respectively) that have been pre-heated to a nominal temperature of 60 °C before exposure with those that have been exposed at 24 °C.
- 3 there is no difference in post-cure mean surface microhardness ratios (bottom/top) for discs of a RC that have been pre-heated to a nominal temperature of 60 °C before exposure with those that have been exposed at 24 °C.

Materials and methods

Spectrum TPH is a light-curing hybrid RC restorative material. The inorganic filler is barium boroaluminosilicate glass (mean particle size < 1 µm), together with colloidal silica (particle size = 0.04 µm). The resin phase comprises a BisGMA adduct with hexamethylene diisocyanate, an ethoxylated bisphenol-A-dimethacrylate,

and triethylene glycol dimethacrylate. Total inorganic filler content is 57% (vol), 77% (mass).

Viscosity

Viscosities (Pa) of two groups ($n = 2 \times 15$) of uncured RC material* were measured using a variable-temperature rheometer[†] in dynamic oscillation mode (frequency = 5 Hz, constant strain amplitude = 5×10^{-4} rad) with a parallel plate configuration, at 60 °C (Group #1) and 24 °C (Group #2). The use of a parallel plate arrangement ensures that the shear rate is relatively constant throughout the material being tested (21). The diameter of the upper parallel plate measured 40 mm. Following heating and dispensation of the RC material onto the lower plate, the upper plate was lowered so that the gap between the upper and lower plates was set at 2 mm. The rheometer was used in continuous oscillation mode with a frequency of 5 Hz. For convenience, readings from the rheometer were recorded at elapsed times after dispensation of 10, 30, 60, 90 and 120 s and compared. Statistical analysis was carried out using SPSS for Windows ([‡] Version 15).

Vickers microhardness (VHN)

The experimental procedure has been reported previously (22), the principal details of which were as follows: Two further groups of RC ($n = 2 \times 15$) were exposed in nylon washers using a QTH LCU[§] at full intensity (nominally 570 mW/cm²) for 30 s. A specimen depth of 1.5 mm was considered to be consistent with the maximum desirable depth for the incremental curing of resin composites (Figure 1). Group #3 was first pre-heated to 60 °C in a dry oven[¶] for 15 min before exposure. Each specimen was then removed from the oven and immediately exposed to the LCU at ambient room temperature (24 °C) with the light-curing tip positioned at 0.9 mm above the specimen surface (which was the thickness of the separating microscope slide) (Figure 1). A reflective background was used under each specimen, which had also been

*Spectrum TPH, Shade C3, Lot 60605212; Dentsply DeTrey GmbH, D-78467, Konstanz, Germany.

[†]CarriMed CSL500; TA Instruments Ltd., Leatherhead, Surrey, UK.

[‡]Version 15; SPSS Inc, Wacker Drive, Chicago, IL, USA.

[§]Degulux Softstart, Dentsply DeTrey GmbH, D-78467, Konstanz, Germany.

[¶]Gallenkamp Hotbox; Sanyo Gallenkamp PLC, Loughborough, UK.

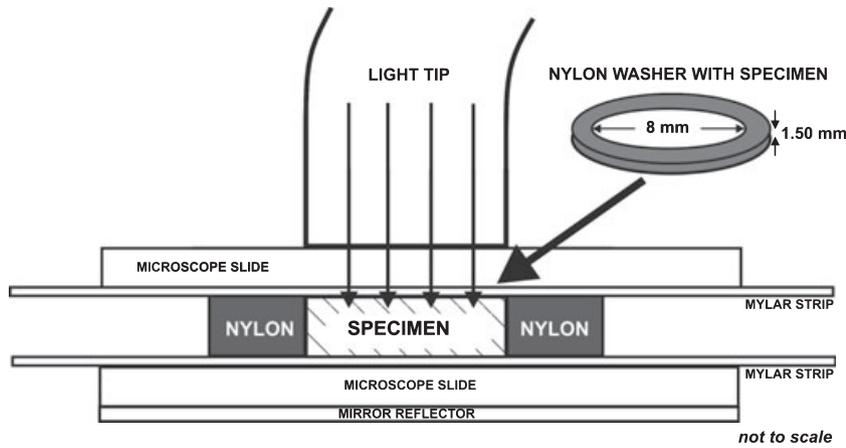


Fig. 1. Schematic of light-curing methodology.

pre-heated to 60 °C. Top and bottom surface VHN were recorded at 24 h after exposure. During this time interval, the specimens were stored dry in an opaque box at 24 °C. Group #4 was similarly exposed and indented at ambient room temperature, 24 °C, without pre-heating.

All microhardness measurements were taken using a calibrated Vickers indenter** using a load of 300 g and a dwell time of 10 s.

Statistical analysis was carried out using SPSS for Windows‡. A repeated measures analysis of variance was used with location (top/bottom) as the repeated measure and Group (#3, #4) as the between-specimens variable.

Results

The experimental results are presented in Tables 1 and 2.

Viscosity

The variability in the two groups was statistically significantly different, so the non-parametric Mann–Whitney test was used to compare the two groups at each time point. A Bonferroni adjustment was made for multiple testing. There was a statistically significant difference ($P < 0.001$) between Group #1 and Group #2 at all times. To investigate the dependence of viscosity on temperature, a linear mixed model was fitted to the

**MVK-H1, Mitutoyo, 1-20-1 Sakato, Takatsu-Ku, Kawasaki-Shi 213, Japan.

data because of repeated observations on the same specimen at different times. At a temperature of 24 °C, the model of viscosity against time is viscosity = 660.2 + 1.09 (time). At a temperature of 60 °C, the model of viscosity against time is viscosity = 261.4 + 1.09 (time). Both time and temperature are statistically significant predictors of viscosity ($P < 0.001$).

VHN

1 There was a statistically significant difference between the measurements taken at the top for Groups 3 and #4 ($P < 0.001$).

Table 1. Mean (SD) viscosity (Pa) of pre-heated and room temperature composite at different time intervals

Time (s)	10	30	60	90	120
Group #1: 60 °C	285 (13)	308 (12)	334 (13)	355 (13)	377 (11)
Group #2: 24 °C	642 (35)	685 (34)	723 (30)	765 (21)	800 (23)

Corresponding data for each time are significantly different ($p < 0.001$).

Table 2. Mean (SD) surface microhardness (VHN) of pre-heated and room temperature composite

VHN	Top	Bottom	Bottom/top ratio
Group #3: 60 °C	68.6 (2.3) ^a	68.7 (1.8) ^a	1.00 ^c
Group #4: 24 °C	60.6 (1.4) ^b	59.0 (3.5) ^b	0.97 ^c

Similar superscripts indicate no significant difference ($p < 0.001$).

2 There was a statistically significant difference between the measurements taken at the bottom for Groups 3 and #4 ($P < 0.001$).

3 There was no significant difference between the measurements taken at the top and bottom for Groups 3 and #4, respectively.

4 There was no significant difference between the bottom/top ratios for Groups 3 and #4.

Discussion

The linear mixed model of viscosity regressed on time indicates a reduction by a mean factor of 2.5 (approximately) for the higher temperature investigated. This significant reduction is in general keeping with previous reports for comparable materials (7, 8) and should facilitate ease of placement and adaptation of the RC material to the internal features of a cavity, thereby reducing the risks of incorporating interfacial voids within the restoration. It may be of interest that the contribution of time to mean viscosity in the linear mixed model is similar for both temperatures.

The VHN data reported for the curing at ambient temperature are comparable to the data reported for the same commercial product and shade with a similar light-curing unit (22). Given the reported correlation between surface microhardness and degree of polymerization, the increased values of VHN for 60 °C may be a reflection of the beneficial aspects of polymerization at a suitably elevated temperature – most likely because of greater mobilities of growing chain moieties in the heated polymerizing material (18). Whether these effects are relevant, *in vivo*, is expected to depend on the rate of ambient cooling and on the time taken in placing the restoration. A recent study reports a 50% temperature drop within 2 min of removing a comparable RC material from a proprietary heating device (23). In the present study, it may be expected that some cooling did occur on removal from the oven but this may not be unrelated to the clinical situation. In contrast, while there might be some concerns regarding placing hot composite adjacent to the vital pulp, it is reported that the major thermal risk is associated with photopolymerization and not with RC temperature (24).

It is of interest that the respective values of VHN for top and bottom surfaces increase in the same proportion in both temperature groups so that a similar bottom/top VHN ratio is maintained (effectively 1.0),

which is in excess of the value of 0.8 suggested for adequate bottom cure (20). However, because of the increased top surface hardness, this has the implication that an improved cure is achieved at the bottom in the heated composite regime, within the parameters investigated. It is likely that a greater depth-of-cure may be achieved on pre-heating (or a similar depth with a reduced exposure), other parameters being equal. This effect is in keeping with a recent report with a comparable material (19).

Within the parameters of the investigation, the null hypotheses are rejected in the cases of temperature effects on viscosity and on respective top and bottom hardness. Thus, it is clear that placement and curing of RC at the elevated temperature should confer the benefits discussed. While the null hypothesis cannot be rejected regarding bottom/top microhardness ratios, this observation is actually beneficial in that a greater depth-of-cure may occur at the higher temperature as a result of the increased top surface hardness observed.

Acknowledgments

The authors acknowledge Dentsply UK for the supply of Spectrum TPH and the Dugulux softstart light-curing unit used in this work.

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