

# Effect of Preheating on Micro-hardness of different Composite Resins

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## Abstract

**Aim:** The aim of this study was to assess the effect of preheating on the surface microhardness of different commercially available composite resins (G-aenial, Filtek Z350 and Tetric-N-Ceram).

**Materials and Methods:** Tubes of the composite resins were either kept at room temperature ( $24 \pm 1^{\circ}\text{C}$ ) or preheated for 40 min in a commercially preheating device to  $40^{\circ}\text{C}$ ,  $50^{\circ}\text{C}$  and  $60^{\circ}\text{C}$ , respectively. From composite resin tubes, a total of 120 disk-shaped specimens (40 specimens of each composite resin, 10 disks from each temperature setting) were prepared by inserting uncured preheated composite resin into cylindrical nylon moulds (5 mm diameter  $\times$  2 mm height); before light polymerized for 40s with a light-emitting diode curing unit. The Vicker's microhardness number (VHN) of each specimen was measured. Two-way ANOVA and Tukey test were used to analyze the collected data at a significance level of 0.05.

**Results:** In comparison to the room temperature, there was a significant increase in the mean VHNs of each composite resin by preheating to  $40^{\circ}\text{C}$ ,  $50^{\circ}\text{C}$  and  $60^{\circ}\text{C}$ , respectively ( $p < 0.05$ ). For G-aenial posterior, Filtek Z350XT and Tetric-N-Ceram, at room temperature, the mean VHNs were  $51 \pm 4.3$ ,  $60.6 \pm 3.7$  and  $76.6 \pm 1.4$ , respectively with significant difference between G-aenial posterior and Tetric-N-Ceram ( $p < 0.05$ ), while at  $40^{\circ}\text{C}$ , the mean VHNs were  $65.7 \pm 2.5$ ,  $86.8 \pm 2.3$  and  $88.4 \pm 1.8$ , respectively with significant differences ( $p < 0.05$ ) between G-aenial posterior and the other two composites. At  $50^{\circ}\text{C}$ , the mean VHNs were  $109.4 \pm 0.9$ ,  $104.4 \pm 3.4$  and  $114 \pm 0.71$ , respectively with no significant difference between composite resins. At  $60^{\circ}\text{C}$ , the mean VHNs were  $122 \pm 3.2$ ,  $129 \pm 3.4$  and  $136 \pm 2$ , respectively with significant difference between G-aenial posterior and Tetric-N-Ceram ( $p < 0.05$ ).

**Conclusions:** Preheating significantly increased the surface microhardness of all tested composite resins with the highest value observed for Tetric N-Ceram at  $60^{\circ}\text{C}$  preheating temperature.

**Keywords:** Composite resins, Preheating, Vicker's microhardness.

## Introduction

Composite resin restorations placement in anterior and in particular, posterior teeth has increased dramatically over the past 5 years.<sup>1</sup> The driving factors for such an increase might be the patient's aesthetic

demand, an increased desire for more conservative restorations and more predictable dental adhesive systems.<sup>2</sup> In posterior teeth, load-bearing composite resin restorations may have a longevity comparable to that of amalgam.<sup>3</sup> However, the use of conventional compositions, such as high-filler content densified or hybrid composite resin materials, may result in poor adaptation to the prepared cavity walls because of high viscosity.<sup>4</sup> In such a framework, the marginal integrity of the final composite resin restoration might be affected because of the subsequent voids entrapment and the microleakage between the composite and the underlying tooth surface.<sup>5</sup> However, composite preheating to a

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temperature of approximately 68 °C has been introduced as a method to increase the initial flow of the high viscous conventional composite resin materials prior to their placement and curing.<sup>6</sup> An improvement in polymerization parameters such as curing depth, reaction rate and degree of conversion was found to be the coincidental aspect of such pre-heating procedure.<sup>7,8</sup> Thus, a positive effect on mechanical properties of composite resin materials might be expected as a consequence to pre-heating, before polymerization, and such an effect has also been reported.<sup>9</sup> Moreover, a mutual relationship has been reported between the degree of polymerization and surface microhardness.<sup>10</sup> Vickers microhardness number (VHN) may be appropriated as a simple monitor of the conversion rate of composite, wear resistance and surface stability of composite, thus it may be identified as being one the best parameters to evaluate mechanical strength. 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.

Chairside warming of composite resin is achieved by placing capsules, or syringes, of the material in a composite warming tray or a water bath. A number of manufacturers present commercial heating devices for this purpose.<sup>11</sup> Calset composite warmer (AdDent, Inc., Dandury, CT, USA) is the most popular and effective preheating device; it can heat the resin to 37°C, 54°C and 68°C, and maintains a constant temperature as needed by the clinician. Another device called Thermo-Flo™ composite warmer has introduced by Vista dental (USA) with a specially designed applicator to keep the material warm for optimal flow and added the advantage of extended length for access in deeper areas.

Warming composite resin has also shown to improved manipulation and resulted in less microleakage<sup>12</sup>, which reduced the film thickness of some conventional materials.<sup>13</sup> Testing the kinetic parameters of resin monomers polymerization revealed an Arrhenius-type behaviour such that a relatively simple increase in temperature may promote a large increase in reaction rate.<sup>14</sup>

The aim of this study was to measure the surface microhardness of three commercially available

conventional composite resin materials (G-aenial, Filtek Z350, and Tetric-N-Ceram) at room temperature and after preheating procedure to 40°C, 50°C and 60°C. These materials are usually used for direct composites posterior restorations. The null hypothesis stated that there is no difference in surface microhardness of the three composite resins at room temperature from that gained after preheating procedure.

## Materials and Method

Three commercially available composite resin materials were used in this study. Tubes of each composite type were preheated as follow, the control tube of each type of composite resin material was kept at room temperature (24°C ± 1°C) without preheating and the other three tubes were preheated to 40°C, 50°C and 60°C in a commercially available preheating device (Figure 1) (Foshan, Stardent, Equipment Co., Limited, China), respectively. Preheated composite resins were used to prepare a total of 120 disk-shaped specimens (40 specimens of each composite resin). The specimens were prepared by inserting the uncured preheated composite resin into the cylindrical nylon moulds (5 mm diameter × 2 mm height). The moulds covered with a Mylar strip and held firmly between two microscopic glass slides (Micro Slides, Gold Seal) on both upper and lower surfaces to remove excess material and standardize surface finishing and to prevent leakage of thermally softened materials, as shown in (Figure 2).

Light was applied to the top surface of each specimen for 40 seconds with a light-emitting diode curing unit (Valo, Ultradent, Cologne, Germany). The light intensity was nominally 1200 mW/cm<sup>2</sup> checked with a radiometer (Patterson curing light meter, Patterson Dental Supply, St. Paul, MN, USA). For the three preheated groups, the composite resin tubes placed in the heating device for 40 minutes before light curing. This time was required for initially warming the device and maintaining the composite resin inside the tube at a constant temperature.

The temperature of each preheated composite material was monitored by inserting a thermocouple probe (1.3 mm diameter) inside the tube of composite resin immediately after removing it from the heating device; the probe fitted to a high-sensitivity temperature recorder (Geratherm medical AG, Gschwend, Germany). Each composite resin material inserted into the mould

within 45 seconds after removal from the heating device. The nylon moulds, clear glass slides, and the plastic filling instrument were warmed to 37°C before insertion of the materials. The composite resin discs stored in a dry opaque box for 24 hours. The surface microhardness analysis was performed with an HVS 1000 digital vickers microhardness tester (Leader precision instruments Co., Shenzhen, China), as shown in Figure 3, using a 100g load for 15 seconds. The microhardness machine provided with Vickers indenter produced a diagonal indentation on the on the top smooth surface of the specimens calculating the VHN at higher magnification. Three randomly selected points for each specimen was measured and averaged.

Statistics performed using SPSS software (SPSS Statistics 17.0, SPSS Inc., Chicago, IL, USA). Two-way analysis of variance (ANOVA) and Tukey test were used for multiple comparisons.

## Results

The preheated composite resin showed higher Vicker's hardness numbers than that of composite resins at room-temperature. Differences in VHNs were significant between the three composite resin materials at each temperature setting ( $p < 0.05$ ), except at 50°C, there was no significant difference in VHNs between the three composite resins. Also, there was continuous increase in VHNs of each composite resin material at different temperatures settings ( $p < 0.05$ ).

At room temperature, the mean VHNs were  $51 \pm 4.3$ ,  $60.6 \pm 3.7$  and  $76.6 \pm 1.4$  for G-aenial posterior, Filtek Z350XT and Tetric-N-Ceram, respectively with significant difference in VHNs between G-aenial posterior and Tetric-N-Ceram ( $p < 0.05$ ). At 40°C, the mean VHNs were  $65.7 \pm 2.5$ ,  $86.8 \pm 2.3$  and  $88.4 \pm 1.8$  for G-aenial posterior, Filtek Z350XT and Tetric-N-Ceram, respectively with significant differences ( $p < 0.05$ ) between G-aenial posterior and both Filtek Z350XT and Tetric-N-Ceram, respectively. At 50°C, the mean VHNs were  $109.4 \pm 0.9$ ,  $104.4 \pm 3.4$  and  $114 \pm 0.71$  for G-aenial posterior, Filtek Z350XT and Tetric-N-Ceram, respectively with no significant difference between composite resins. At 60°C, the mean VHNs were  $122 \pm 3.2$ ,  $129 \pm 3.4$  and  $136 \pm 2$  for G-aenial posterior, Filtek Z350XT and Tetric-N-Ceram, respectively with significant difference between G-aenial posterior and

Tetric-N-Ceram ( $p < 0.05$ ).

## Discussion

Based on the results reported here, the null hypothesis, stating that there is no significant correlation between pre-curing warming of composite resin and post-curing microhardness should be rejected. The three types of composite resin tested in the current study showed increased values of microhardness as a direct response to pre-curing warming. For all the three resin composites used in the current study, surface microhardness increased with preheating, these findings are in accordance with the results of previous studies.<sup>11, 15-18</sup>

In order to explain these results, it is important to understand that during polymerization, as soon as exposure to light, the process of monomer conversion will be initiated. With the progression of the reaction, composite will become more viscous with the formation and growing of the polymer chains. The increased in viscosity will limit the movement of molecules at this vitrified state, and prevents the completion of the polymerization.<sup>14, 19</sup> Contrariwise, as a result of higher thermal energy, preheated composites exhibit an increase in monomer mobility, reducing the viscosity and augmented the molecular motion.<sup>15, 19, 20</sup>

Studies showed that, light curing of warmed composite resin even for a shorter period of time demonstrated a higher degree of conversion than light-curing at room temperature.<sup>8</sup> On the other hand, refrigerated composite resin illustrated lower conversion rate.<sup>14</sup> The increased viscosity of the refrigerated material resulted in slower propagation and subsequently decreased in monomer conversion. A correlation between monomer conversion and surface microhardness values has been reported in several studies.<sup>9, 17, 21, 22</sup> In the current study, this might explain the significant rise in surface microhardness among all tested composite resin materials. It is important to note that all composites tested were light activated type, the polymerization reaction is initiated by light only, pre-curing warming of composite resin showed no evidence of polymerization until the material exposed to light.

Dental composites can be defined three-dimensionally by filler, organic matrix and coupling

agent. The hardness of composite resins reflects their molecular chain flexibility and degree of polymerisation<sup>9, 17</sup> and is affected by other factors such as resin matrix type, filler type and filler volume fraction. According to the results of this study, the chemical composition of each composite may have a significant effect on the microhardness of each tested material. Tetric-N-Ceram resin composite exhibited the highest mean microhardness under the various experimental conditions investigated, followed by Filtek Z350XT and G-aenial posterior. The high microhardness values achieved by both Tetric-N-Ceram and Filtek Z350XT resin composites may be related to the amount and type of fillers. These materials have high filler contents (63.5%, and 78.5% by weight, respectively) and employ the nano-filler technology, no significant difference was found between these two materials in surface microhardness. Filler content has been also correlated with depth of polymerization, hardness, compressive strength, and stiffness<sup>23</sup>. The presence of fillers enhances the resistance of the composite to abrasion, improves its mechanical strength and resistance to indentation.

The presence of different filler particle size could be another factor that affects the microhardness. As the light beam is scattered and reflected within the composite material during light-curing this can affect the degree of composite conversion, leading to lower microhardness values. Larger filler size variation in G-aenial posterior (16-200nm) might explain the lesser microhardness value when compared with Filtek Z350XT (4-20nm) and Tetric-N-Ceram (40-160nm).

### Conclusion

within the limits of this study, warming of the resin composite materials significantly increased their surface microhardness; the greatest increase occurred at 60°C, which may be recommended as suitable preheating temperature for all materials tested. This recent trend in composite application is promising, improving the mechanical properties, and thus enhance the durability of the restorations.

**Financial Disclosure:** There is no financial disclosure.

**Conflict of Interest:** None to declare.

**Ethical Clearance:** All experimental protocols were approved and all experiments were carried out in accordance with approved guidelines.

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