

**Comparison of Post-Polymerization Microhardness Values for a
Representative Bulk Fill Composite Placed Using Room-
Temperature and Preheated Placement Regimens**

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TABLE OF CONTENTS

ACKNOWLEDGMENTS.....	4
COPYRIGHT.....	5
ABSTRACT.....	6
INTRODUCTION.....	6
MATERIALS AND METHODS.....	8
RESULTS.....	9
DISCUSSION.....	9
CONCLUSION.....	11
REFERENCES.....	13
APPENDIX.....	14

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ABSTRACT

Purpose: The objective of this study is to see if the preheating of bulk fill resin composite increases its microhardness physical properties compared to room temperature bulk fill resin composite.

Methods: Two Groups of Filtek™ One Bulk Fill Composite were evaluated: one at room temperature and another preheated in a BioClear HeatSync Composite Warmer Kit to 155°F. The composites were then injected into fifteen 4.5 mm discs and cured for 20 seconds. The discs were placed on an Instron LM300 AT tester and using a Knoop hardness test evaluated at 4 separate points on the top and bottom of the discs. The data was analyzed and reviewed for statistical analysis.

Results: Examination of the overall Knoop Hardness Number (KHN) of the two groups showed significant effects of temperature on microhardness. The overall average of the room temperature had a mean hardness of 29.1 KHN compared to an overall hardness of 34.4 KHN with bulk fill composite at heated temperature. In both groups, the top side of the cure was significantly harder than the bottom side of cure.

Conclusions: Preheating Filtek One Bulk Fill composite shows an increase in microhardness after polymerization.

Keywords: composites, bulk fill, preheated composite, microhardness

Introduction

Since their introduction in the 1960s, resin-based composites have assumed increasing importance in restorative dentistry[1]. Dental composites consist of: highly cross-linked polymeric resin matrices; glass, silica, or crystalline filler particles; silane coupling agents; and initiators which are central to polymerization of the resin mass[2]. Currently, composite resins are used as direct and indirect restorative materials, bonding agents, and dental sealants[2].

Dental composites offer many advantages within the restorative arena. Their esthetic properties (i.e., color, translucency, etc.) make them suitable for use in anterior applications where esthetics are critical. Composite resins also display bonding characteristics which permit improved retention, decreased microleakage, and conservative tooth preparation[3]. Furthermore, composite resins have lower thermal conductivities than their metallic counterparts [3], so patients experience less sensitivity.

Despite the favorable characteristics displayed by composite resins, these materials exhibit limited depth of cure and clinically significant polymerization shrinkage. Since the curing process of the composite materials are initiated by light waves, the light waves must reach the bottom of the preparation, to avoid having uncured material at the base. This uncured, or partially cured, material will be more prone to fracture and at increased risk of gap formation, resulting in food and bacteria traps[3]. This has been avoided by incremental buildup of the restoration in smaller layers, curing every 2 mm to make sure all composite material is properly set.

Another way to prevent polymerization shrinkage and ensure composite is adequately cured is the use of bulk fill composite. By manipulating the refractive indices and filler size particles, manufacturers have made a more translucent composite material, allowing light to penetrate deeper (4-5 mm) and result in a better cure[4]. This process also allows the procedure to take

less time and a deeper cure reduces the risk of developing voids[4 5].

When working with Bulk Fill Composites, the high viscosity will make it difficult to adapt and adhere to the tooth. Many practitioners have turned to preheating the composite to overcome this issue[6 7]. Research by Deb et al has shown that prewarming composite decreased film thickness, resulting in a more viscous material[8]. Studies with other types of composite have also shown better marginal adaptation[6 9 10], increased monomer conversion[6 11-14], and enhanced surface hardness when preheated[7 15]. However, studies have shown when preheating composite, delaying its use can have negative effects on the material's physical properties[10 12 16].

So how will heating bulk fill composite effect its physical properties? A new heating device, the HeatSync Composite Warmer Kit (Bioclear; Tacoma, WA; USA) heats composite up to 155 degrees Fahrenheit to allow better flow through preformed matrix bands. Will heating the composite at such a high temperature have any effect on the hardness of the cured material? The purpose of this study was to determine if preheating a bulk fill composite material with the BioClear HeatSync Composite Warmer Kit would have any effect on the microhardness of cured composite compared to composite cured at room temperature.

Materials and Methods

The Knoop Microhardness test was used to assess the toughness of 30 samples of Filtek™ One Bulk Fill composite resin (3M™ Oral Care Solutions Division; St. Paul, MN; USA). Shade A2 was selected to make 15 samples of each group. Samples were split evenly between two groups based on temperature. One group was cured at room temperature and one group was preheated to 155 degrees Fahrenheit with the HeatSync Composite Warmer Kit (Bioclear; Tacoma, WA; USA) (Figure 1) prior to pouring into the mold. Four and half millimeter molds

were used and the height of the molds were verified using a digital caliper (iGAGING, San Clemente, CA; USA). Each sample was cured for 20 seconds each with an Elipar DeepCure-S LED cure light (3M™ Oral Care Solutions Division; St. Paul, MN; USA).

The Scrape test was performed on the bottom surface of the samples, removing any uncured resin. All the samples were then tested in a Instron LM300 AT tester (Leco, LM300 AT, St Joseph, MI; USA) (Figure 2) under a load of 50 grams for a dwell time of 15 seconds. For each sample, a Knoop Hardness Number (KHN) was obtained by dividing the applied load (kg) by the projected area of the impression (mm²). Knoop hardness number (KHNs) were collected from four sites on both the top and bottom surfaces. Thus, for each sample, a total of eight measurements were taken resulting in a total of 240 KHNs.

Statistical Analysis

Normality of the KHN sampling distribution was confirmed with the Shapiro-Wilk test and assumption of homogeneity of variance was assessed using Levene's test. Sample hardness measures are reported as means with associated standard deviations. Knoop hardness ratios were obtained by dividing the bottom side hardness of each sample by its respective top side hardness. Statistical analysis was performed using a one-way analysis of variance (ANOVA). Eta squared (η^2) values are provided for significant results as measures of effect size. Data were analyzed using SPSS version 25 (IBM, Armonk, NY, USA). Statistical significance for all statistical tests was declared at $P < 0.05$.

Results

Examination of the overall KHNs of the two groups revealed a significant effect of temperature ($P < 0.001$, $\eta^2 = 0.17$). The samples cured at room temperature had a mean hardness of 29.1 (SD = 6.0) compared to 34.4 (SD = 5.8) for the samples which were pre-heated.

Similarly, when assessing the differences between the two groups based on sample location, similarly higher KHNs were observed for both the top and bottom sides of the pre-heated samples (top $\eta^2 = 0.68$, bottom $\eta^2 = 0.70$).

In both groups, the top side was significantly harder than the bottom (both $P < 0.001$). The bottom to top KHN ratio was 0.67 (SD = 0.07) for the room temperature samples and 0.72 (SD = 0.05) for the pre-heated samples, $\eta^2 = 0.14$. Mean KHNs for each group are shown in Table 1 and Figure 3.

Discussion

The results showed that by increasing the temperature of the composite, we increased the microhardness of the material, thereby allowing us to accept our hypothesis.

The Knoop Microhardness Values measured from the top of samples had overall higher values compared to measurements made at the bottom of the sample. This can be explained by the proximity of the light source to the material. The composite hardening is initiated by light waves, so the closer the material is to the light source, the more light waves it is able to absorb, polymerize, and harden. The light source used in this experiment is a 3M ESPE Elipar DeepCure-S cure light. Based on the instruction manual, the light has a collimated, uniform light beam profile, allowing the light to be evenly distributed down the column[17]. Studies within the cure light manual showed that at a specimen depth of 4.5 mm, Vickers hardness test for multiple restorative materials studies dropped[17]. With Bulk Fill composite, hardness dropped from 100% at 0.5 mm to around 97% at 4.5 mm[17]. These findings also correlate with the findings of Zorzini et al, when testing hardness of bulk fill resin composite, his team found that hardness was reduced from the top of the source to the bottom[18].

The main focus of the experiment showed that heating the composite showed greater

microhardness measurements at both the top and bottom of our sample readings. One reason for increase in microhardness is the increase in amount or degree in polymerized material. Calherios and Daronch both saw increase in degree of polymerization when increasing the temperature of the material[12 13]. With an increase in polymerization, it would result in increase in cured/hardened material, thereby improving the hardness. In a separate article by Daronch, heating the material is believed to increase the mobility of composite molecules and free radicals, increasing the chance of them contacting and interlinking with each other, resulting in increased crosslinking and stronger bonds[19].

Future Studies

Future studies can evaluate different perspectives in the light cure procedural process of composite transformation. This study focused on one type of composite warming device. Studies can be done evaluating the different devices currently on the market and over ways of warming composite material (water bath, placement by a light source) to see if that has any effect on the physical properties. Also, the temperature used in this experiment was set to 155°F. Would increasing the temperature beyond that set in this experiment result in greater microhardness values and at what temperature would see no change or is there a temperature where we can see a negative change. Also, the temperature set for this material was used for cavity preparations set in enamel. For deep restorative preparations, will the temperature of the composite cause pulpal irritation? Another aspect that could be evaluated is the cure time. Can we decrease the cure time of the material but maintain the same microhardness? By decreasing the cure time, we lessen the exposure of a heat source to the tooth, lessen the risk of causing pulpal irritation.

The main limitation for this study was the use of only one type of bulk fill composite material. By looking at multiple brands of bulk fill, we could get a better understanding if the manufacturer has any effect on the physical property results. Also, packable bulk fill composite was only studied. There are flowable bulk fill composite brands that could be tested as well to see if the microhardness is affected by heat.

Conclusion

Within the limitations of this study, it can be concluded that preheating the composite material resulted in much higher microhardness readings compared to the composite cured at room temperature. It was also shown that the hardness values measured closer to the light source were higher than those hardness values further from the light source. Based on this data, further studies can be conducted studying different composite materials and heat sources and how that affects their physical properties.

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Appendix

Figure 1: BioClear HeatSync Composite Warmer Kit



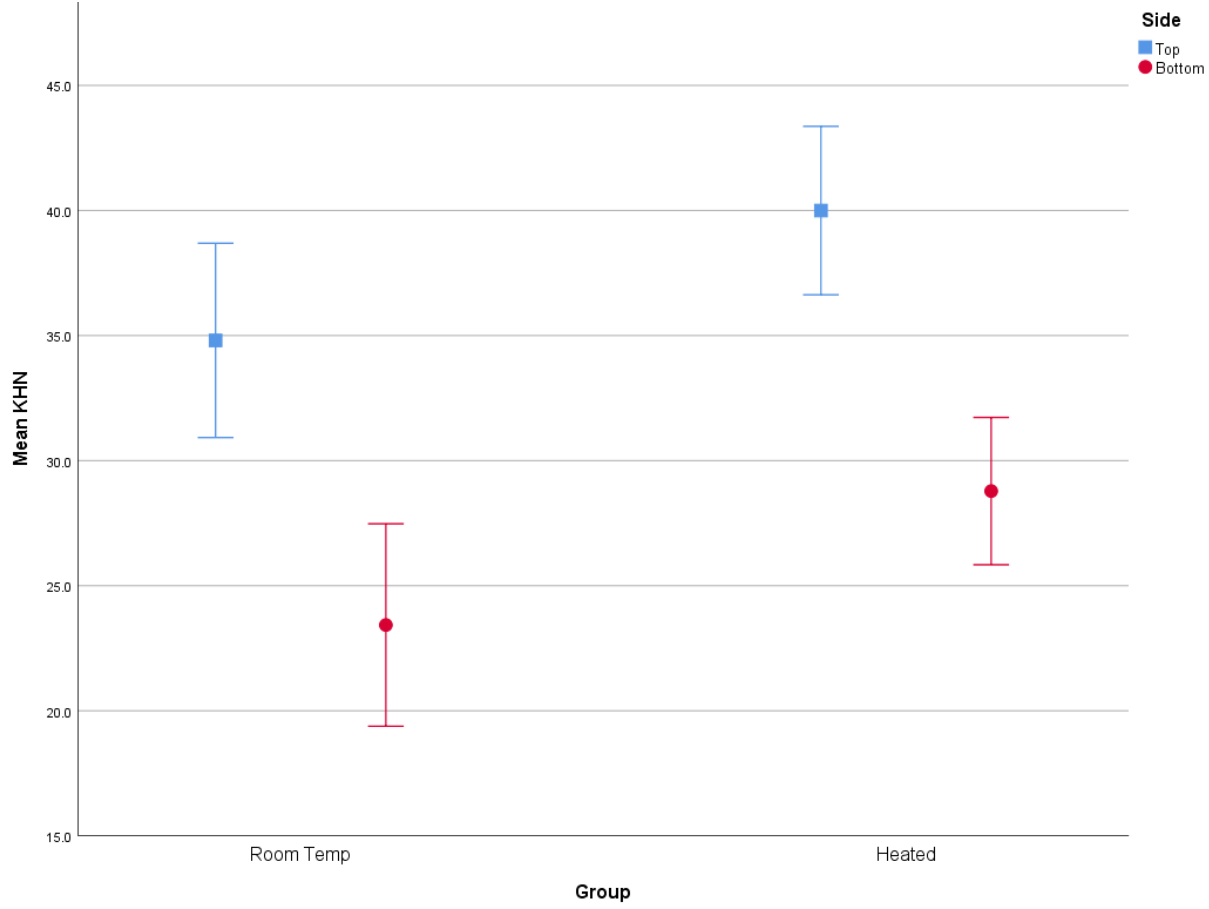
Figure 2: Instron LM300 AT tester



Table 1. Knoop Hardness Number by Group and Sample Location

	Room Temp		Heated		P	η^2
	Mean	SD	Mean	SD		
Top	34.8	1.9	40.0	1.7	<0.001	0.68
Bottom	23.4	2.0	28.8	1.5	<0.001	0.70
Total	29.1	6.0	34.4	5.8	<0.001	0.17

Figure 3. Mean Hardness by Group and Sample Location*



*Error bars represent 2x standard deviation