



Effects of Different Temperatures and Storage Time on the Degree of Conversion and Microhardness of Resin-based Composites

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ABSTRACT

Objective: Dental materials are often made at room temperature, whereas clinically they are made in the mouth. This study evaluated the effects of temperature on the degree of conversion (DC) and Knoop microhardness (KHN).

Materials and methods: Two types of resin-based composites (RBCs) were light-cured using a light-emitting diode (LED) light-curing unit. The resin specimens were centered on an Attenuated Total Reflectance Fourier transform infrared (FT-IR) plate heated to 23°C or 33°C. The DC of the resin was calculated after 120 seconds, the specimens were removed, and the KHN was tested at the bottom of the specimens both immediately, after 24 hours, and after 7 days storage in distilled water in complete darkness at 37°C. The effects of different temperatures on the DC and KHN with their storage time were compared by analysis of variance and Fisher's protected least significant difference *post hoc* multiple comparison tests ($p < 0.05$).

Results: Increasing the temperature had a significant and positive effect on the DC and KHN for immediate values of the RBCs. Greater conversion and hardness occurred when the curing temperature was increased from 23°C to 33°C. The KHN increased significantly after 24 hours of storage. There was a linear relationship between DC and KHN ($R^2 = 0.86$) within the range of DC and KHN studied.

Conclusion: The physical properties of dental materials can be expected to be better when made in the mouth than when they are made in a laboratory at room temperature.

Keywords: Bulkfill RBC, Degree of conversion, KHN, Temperatures.

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INTRODUCTION

Over the last three decades, light-cured resin-based composites (RBCs) have become the most prevalent direct dental restorative material.¹ During that time, the esthetics and both physical and mechanical properties of RBCs have improved.

The placement of a successful RBC restoration is a technique-sensitive procedure. It is important that the clinician know the features and the requirements of the RBC they are using to deliver an adequately cured restoration. In general, conventional light-cured RBCs are placed and light cured in 2-mm increments.² However, manufacturers now claim that their new bulk-fill RBCs can be placed and adequately cured in 4-mm or even 6-mm increments.³

Resin-based composites can be light-cured with different types of light-curing units (LCUs). Currently, the light-emitting diode (LED) curing unit is the most commonly used in dental practice.⁴ Contemporary LED units often deliver high irradiance values above 2000 mW/cm² and offer a reduction in the light exposure time. This use of these high-power LCUs, together with the use of bulk-fill RBCs, can result in a significantly large time saving when placing resin-based restorations.

However, clinicians can easily miscalculate the time required for adequate light-curing of each increment of

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an RBC, or the thickness of the RBC they are placing. This may then result in an inadequate amount of light delivered to the RBC and this will create an inadequately cured RBC. This is highly undesirable because this will lead to reduced physical and mechanical properties, increased bacterial colonization, and leaching of greater amounts of uncured monomer into the body.^{5,6} Conversely, excessive light-curing (especially with high-power LCUs) may overheat the soft and hard tissues, leading to possible pulpal irritation or soft-tissue burns.⁷

The dental literature includes reports from many laboratory experiments that have evaluated RBCs. Tests that evaluated the degree of conversion (DC) and microhardness are considered to be valid and reliable. However, most of these tests, including the ISO 4049 depth of cure standard, were conducted at room temperature, which is not the same as the temperature in the oral cavity.

It is well-known that temperature has an effect on the rate of polymerization and the DC of an RBC, leading to possible alterations in its physical and mechanical properties. As the temperature increases, resin viscosity is reduced and molecular mobility increases, all leading to increases in the DC,^{8,9} but this effect appears to have been overlooked in many studies. The effect of testing RBCs at the same temperature found in the tooth will provide more appropriate values for the materials and help avoid either over- or undercuring of the RBC increments.

Several studies have investigated the effect of temperature on resin-curing. In a 1999 study, Lovell et al¹⁰ examined experimental resin and found an increase in the DC when increasing their tested temperatures (25°C, 40°C, and 55°C). Other studies evaluated different commercial resins and arrived at similar conclusions. In 2004, Trujillo et al¹¹ found a clear relationship between temperature and resins' DC. In their study, they tested a material's DC at different temperatures (23°C, 37°C, 54°C, and 70°C) and concluded that the higher the temperature used, the higher the percentage of resin converted.¹¹ Other studies showed an increase in shrinkage strain rate when temperature increased.^{12,13} A recent study by Watts and Alnazzawi¹⁴ confirmed the previous studies' findings, wherein there was an increase in the shrinkage stress and stress rate values of tested resins when temperature changed from 23°C to 37°C. However, temperatures above

37°C are not found intraorally. Another recent study evaluated the effect of temperature evaluation on KHN and DC on a 2-mm-thick RBC. Price et al¹⁵ reported a significant difference in DC (47.6% at 22°C and 57.8% at 35°C) and KHN (21.4% at 22°C and 31.8 at 35°C).

The purpose of this study was to evaluate the effects of a simulated intraoral temperature and three measurement times on DC and KHN of two RBCs. The null hypotheses were:

- There would be no difference in DC and KHN values for RBCs when cured at room temperature compared with a simulated intraoral temperature.
- There would be no difference between immediate and postcuring readings (24 hours and 7 days) for the KHN values of RBCs.
- There would be no difference in DC and KHN values at the bottom of 2 and 4-mm thicknesses of RBCs.

MATERIALS AND METHODS

Two types of RBC were used, an IVA shade of Tetric EvoCeram Bulk Fill (Ivoclar-Vivadent, Amherst, NY) and a A2B shade of Filtek Supreme Ultra (3M ESPE, St Paul, MN). All specimens were light-cured using a Valo LED LCU (Ultradent, South Jordan, UT) (Table 1) with the light tip placed directly over each specimen.

Spectral Radiant Power and Radiant Exposure Received by the Specimens

The irradiance, radiant exposure, and spectral emission from the LCU were measured by means of a 6-inch integrating sphere (Labsphere, North Sutton, NH) connected to a fiber-optic spectrometer (USB 4000; Ocean Optics, Dunedin, FL). This fiber-optic system was calibrated before the experiment using the internal reference lamp contained within the sphere. The output from the LCU was measured through a 4-mm diameter aperture placed over the entrance to the integrating sphere. This aperture matched the 4-mm diameter of the specimens, so that the sphere measured the same spectral radiant power that would be received by the specimens and not the total output emitted from the LCU. Spectrasuite v2.0.162 software (Ocean Optics, Dunedin, FL) was used for data collection and analysis. So, that the radiant exposure received by the 4-mm diameter

Table 1: The resin-based composite resins used in the study, along with their curing light units

Material (shade)	Abbreviation	Type	Manufacturer	Lot #
Filtek Supreme Ultra (2AB)	FS	Conventional	3M ESPE, St Paul, MN, USA	N553090
Tetric EvoCeram Bulk Fill (IVA)	TBF 2 or 4 mm	Bulk Fill	Ivoclar Vivadent, Amherst, NY, USA	R08233
Valo		LED	Ultradent, South Lake, UT, USA	V37330
Elipar S10		LED	3M ESPE, St Paul, MN, USA	939112-01277
Bluephase G2		LED	Ivoclar Vivadent, Amherst, NY, USA	P634762 S607644

specimen in 10 seconds could be determined, the area under the power-*vs*-time graph was calculated.

Specimen Fabrication

The Filtek Supreme Ultra RBC was expressed and packed into a 2.0-mm thick metal ring, whereas the Tetric EvoCeram Bulk Fill was expressed into both 2.0 and 4.0-mm thick metal rings (Fig. 1). Both rings had a 4.0-mm internal diameter that matched the mold diameter as specified in the ISO 4049 standard and the 4.0 mm aperture into the integrating sphere. The RBC specimens were centered on the diamond element of a horizontal attenuated total reflectance (ATR) attachment (Golden Gate, SPECAC, Inc., Smyrna, GA) in a Fourier transform mid-infrared (FTIR) spectrophotometer (Bruker Optics Inc., Billerica, MA). The top surfaces of the uncured samples were covered by a Mylar strip to avoid contact with the curing light tip. Each RBC sample was exposed to light for 10 seconds using the Valo in the standard curing mode.

Degree of Conversion and Knoop Microhardness Measurements

The DC was collected in real time for 120 seconds at two different temperatures, 23°C and 33°C, on the ATR plate (Fig. 2). The spectrometer data collection rate was 8.4 measurements per second. Data were collected for 10 seconds before the LCU was switched on to determine the DC baseline of the uncured RBC, and for 110 seconds thereafter.

The RBC specimens were irradiated for 10 seconds according to the digital timer of the LCU. After the DC at the bottom had been collected for 120 seconds, the specimen was removed and placed immediately on a Knoop microhardness testing device (HM 123; Mitutoyo Canada Inc., Mississauga, ON, Canada).

The microhardness was measured as soon as physically possible (within 5 minutes for bottom and within 15 minutes on top surfaces) at nine points, first across the bottom surface within the region where the DC measurements were made and then at nine points across the top surfaces of each specimen.

Evaluation of the Test Results

Five specimens were made for each condition. After the hardness measurements were made, the specimens were stored in distilled water in complete darkness at 37°C. Knoop hardnesses were retested 24 hours later and again 7 days after the samples were made. The mean bottom KHN and mean top KHN values were compared to determine the bottom:top hardness ratio.

The effects of the different temperatures on the DC and immediate KHN values, and the effects of 24-hour and 7-day storage on KHN, were compared by three-way analysis of variance (ANOVA) and Fisher's protected least significant difference (PLSD) *post hoc* multiple comparison tests ($p < 0.05$).

RESULTS

Based on the power-*vs*-time data, the RBC specimens received a mean irradiance of 1450 mW/cm² for 10 seconds and a radiant exposure of 14.5 J/cm² (Graph 1).

The DC values for the specimens cured at 23°C were lower than those for specimens cured at 33°C (Graph 2). This was shown for both RBC types tested at different depths.

The KHN was also lower for the specimens cured at 23°C than those cured at 33°C ($p < 0.05$). The KHN increased significantly at 24 hours but not at 7 days [except for the bottom surface of TBF 4 mm at 23°C] ($p < 0.05$) (Tables 2 and 3). For the TBF2, KHN value increased by 26.1% between curing at 23°C and 33°C

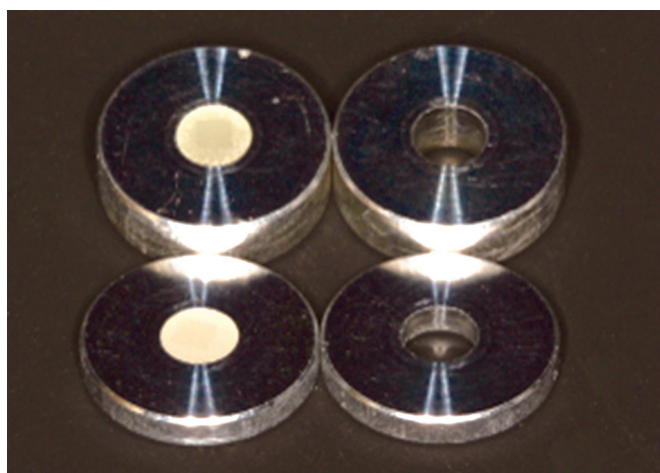


Fig. 1: Metal rings with and without cured RBC. Rings were 2 or 4 mm thick with an internal diameter of 4 mm

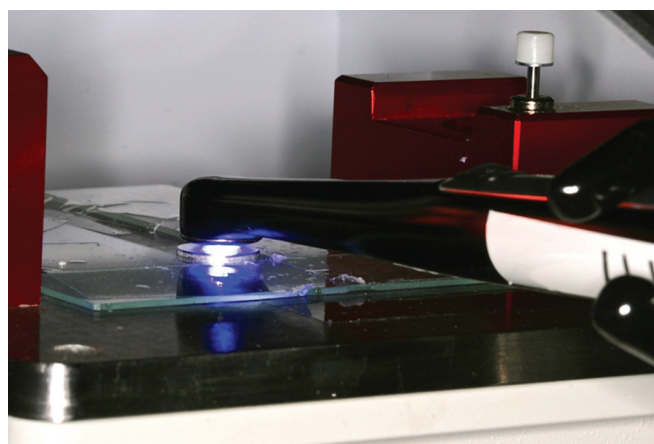
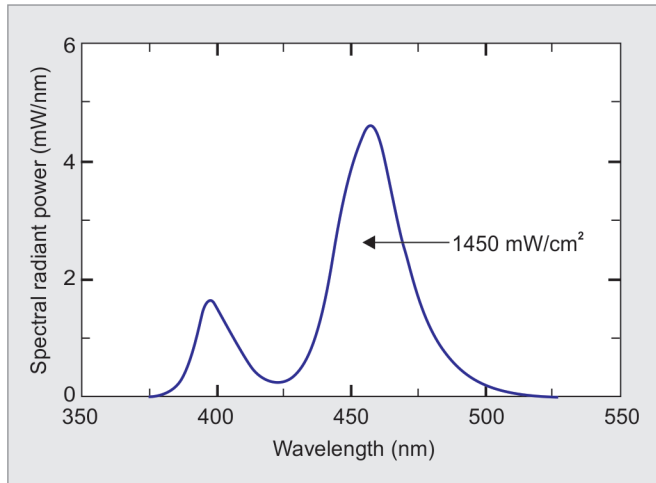
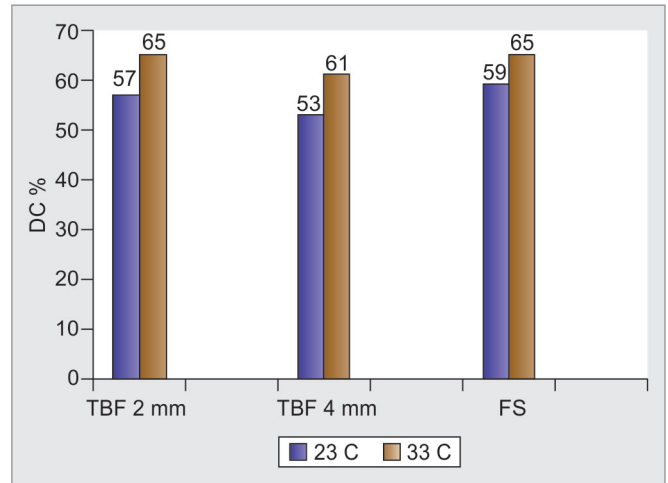


Fig. 2: Valo LED fixed above RBC during light curing. Note that the metal ring (with cured resin) is centered on the diamond element of the heated ATR attachment in an FTIR spectrophotometer



Graph 1: Spectral emission from the Valo measured through a 4-mm aperture into an integrating sphere, showing two distinct emission peaks



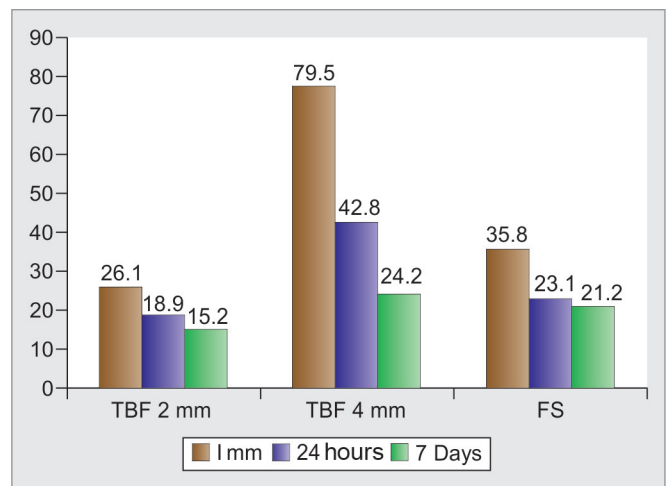
Graph 2: Degree of conversion of RBCs at temperatures of 23°C and 33°C

13.8–17.4), whereas for the same thickness, FS showed a 35.8% increase (21.2–28.8). The TBF4 samples showed a highly significant increase of 79.5% (7.5–13.5). At the top of the samples, TBF2 had an 8.4% increase, and FS had an 18.2% increase, but TBF4 showed the greatest increase in KHN (27.3%) (Graph 3).

The bottom:top hardness ratio for all samples cured at 33°C was greater than that for samples cured at 23°C. For example, at a temperature of 23°C, TBF2 had 62.6%, FS2 had 69.4%, and TBF4 was only 36.4%, but at 33°C, samples of TBF2, FS, and TBF4 were 72.8, 79.6, and 51.3% respectively.

DISCUSSION

Previous studies have reported that higher temperatures from 40°C to 60°C will have a strong and positive influence on the polymerization kinetics of a dental restorative composite.^{9,14} This study showed that a lower



Graph 3: Differences in percentages for immediate, 24-hour, and 7-day KHN values for the three RBCs at temperatures of 23°C and 33°C

Table 2: Means and standard deviations (±SD) [top (T) and bottom (B)] for immediate and delayed (24 hrs and 7 days) KHN for RBCs, Tetric EvoCeram Bulk Fill, 2 mm (TBF2) and 4 mm (TBF4) thick, and Filtek Supreme Ultra, 2 mm thick (FS) at 23°C

Temp.	23°C					
	TBF2		TBF4		FS	
	T	B	T	B	T	B
Immediate	22.04 (0.50)	13.80 (1.37)	20.66 (0.81)	7.52 (0.50)	30.62 (1.20)	21.24 (1.05)
24 hrs	25.14 (1.14)	16.38 (0.43)	24.92 (0.61)	10.50 (0.83)	35.30 (1.20)	25.12 (0.86)
7 days	25.12 (0.62)	17.12 (1.62)	25.10 (0.84)	12.36 (0.27)	35.34 (1.62)	26.46 (0.53)

Table 3: Means and standard deviations (±SD) [top (T) and bottom (B)] for immediate and delayed (24 hrs and 7 days) KHN for RBCs, Tetric EvoCeram Bulk Fill, 2 mm (TBF2) and 4 mm (TBF4) thick, and Filtek Supreme Ultra, 2 mm thick (FS) at 33°C

Temp.	33°C					
	TBF2		TBF4		FS	
	T	B	T	B	T	B
Immediate	23.9 (0.77)	17.4 (0.95)	26.3 (1.42)	13.5 (0.94)	36.2 (1.66)	28.8 (1.36)
24 hrs	26.3 (0.59)	19.5 (0.74)	28.4 (1.16)	14.9 (0.72)	38.2 (0.96)	30.9 (1.10)
7 days	26.8 (0.55)	19.7 (1.0)	28.8 (1.75)	15.4 (1.29)	38.5 (0.65)	32.0 (1.40)

and more clinically relevant temperature also has a highly significant effect on the DC and KHN values of both conventional and bulkfill RBCs. Thus, the first hypothesis was rejected. This effect could be seen in the immediate, 24-hour, and 7-day samples. Thus, the second hypothesis was rejected. There was also a significant difference between the results from the 2 and 4-mm thick specimens of RBCs; thus, the third hypothesis was rejected.

Many studies, including the ISO standard, have investigated the properties of RBCs at room temperature (20°C–24°C).^{8,16,17} However, the value of such tests when these RBCs are at a higher temperature (i.e., in the mouth) must now be questioned. This study showed that higher DC and KHN values were reached on all the surfaces of specimens tested when cured at 33°C. This occurs because, at higher temperatures, there is an increased molecular mobility, leading to an increase in monomer conversion.^{8,14} As the temperature increases, the diffusion-controlled propagation, the reaction diffusion controlled termination, and the autodeceleration phases are delayed, thereby allowing the RBCs to reach higher DC values before vitrification. As a result, a more highly crosslinked polymer network with improved mechanical and physical properties is produced at higher temperatures. This effect is more pronounced as the thickness of the RBC increases.^{9,18,19}

For any RBC to be approved for commercial use, its mechanical properties (e.g., microhardness) must be evaluated. Testing protocols for dental resin materials involve ISO standard 4049.²⁰ To comply with ISO test methods, the RBCs used in this study were prepared in a metal mold with an internal diameter of 4.0 mm, the DC was measured during curing, and the KHN was tested soon after light-curing was completed.

Our findings showed lower bottom:top hardness ratio percentages when compared with others reported in the literature.^{21–23} Although specimens involved in this study received energy of 14.5 J/cm², our immediate values were in the range of KHN 36.4 to 69.4 and a bottom:top hardness ratio of 51.3 to 79.6% when cured at 23°C and 33°C respectively. Some previous studies reported in the literature have used a 6 or 8-mm diameter mold when testing dental materials. A recent study by Erickson et al²⁴ examined the effects of different mold diameters on the DC of one RBC. They tested the effects of using molds that were 3, 4, 5, and 6 mm in diameter and found that as the diameter decreased, the DC also decreased. This occurred because the relative absorption of the curing light by the walls of the mold was greater as the volume of the RBC was reduced. Thus, less light energy reached the bottom of the RBC, and the DC was lower at the bottom.^{24,25} These previous findings, together with others reported in the literature,^{26,27} could explain why this experiment showed lower values regarding bottom hardness values for the RBC samples in this study.

The effect of time on the RBCs was the same for all specimens. All had an increased KHN after 24 hours compared with their immediate values (Tables 2 and 3). At the top surfaces of the samples cured at 23°C, there was a similar small increase after 24 hours of storage that ranged between 14.1 and 20.6% for both RBCs. The bottom surface showed a greater increase. The increase in TBF2 and FS was 18.7 and 18.3% respectively, but for TBF4, the increase jumped to 39.6%. In contrast, all the samples cured at 33°C had a similar percentage of increase. At the top surface, there was a small increase of 10, 5.5, and 8.6% for TBF2, FS2, and TBF4 respectively. At the bottom surface, there was a similar small increase that ranged between 7.3 and 12.1%.

In comparisons of KHN values after 24 hours for samples cured at 23°C and 33°C, there was a clear difference. At the top surfaces, the KHN for TBF2 and FS showed a small increase of 4.6 and 8.2%, but TBF4 had a larger increase of 14%. The KHN values at the bottom surface also increased. The largest increase was seen in the TBF4 (41.9%). The TBF2 and FS had similar smaller increases of 19 and 23% respectively. This effect continued in the same pattern when the samples were analyzed after 7 days of storage. At the top surface, the increase in KHN values ranged between 6.7 and 14.7% [6.7 (TBF2), 8.9 (FS), 14.7 (TBF4)]. For the bottom surfaces, the percentage ranged between 15.1 and 24.6%. These results suggest that temperature has a greater effect when the RBC is less well-cured, that is, has a lower DC, and supports previous findings reported by Daronch et al.⁹ The results also showed that even after 7 days, the samples cured at 33°C still had higher values in both top and bottom surfaces.

In this study, the correlation between DC and KHN was also evaluated. After DC measurements were collected, the study protocol minimized the time difference between when the DC and the KHN values at the bottom were recorded. There was a good linear correlation between DC and KHN at both temperatures [$R^2=0.86$ (23°C) and $R^2=0.82$ (33°C)]. This is in contrast to studies that reported a weaker correlation. The better correlation probably occurred because of the minimal time difference between the DC and the KHN measurements, and the RBCs were not polished. Polishing the specimen would have taken time and may create a harder and better cured surface due to heat generation.

This study showed that testing RBCs according to the ISO 4049 standard likely simulates the “worst case scenario.” Due to the lower bottom KHN values found in this study, and to rule out any potential issues because the Valo curing unit was used, the LCU from the manufacturer of the RBC materials was also used to prepare a few specimens of RBC for comparison. An

Elipar S10 (3M ESPE, St Paul, MN) was used to cure FS, and the Bluephase G2 (Ivoclar Vivadent, Amherst, NY) was used to cure TBF. Each sample was cured at 33°C (total of three samples for each FS, TBF2, and TBF4). The results showed that when these RBCs were cured in 4-mm diameter metal molds for 10 seconds, comparable results were obtained. These lights were used for 60 seconds, when they produced higher bottom KHN values (with a bottom/top ratio of 86 to 90.5%).

This study used only two RBCs, but in light of results reported from other studies, it is probable that other RBCs will also achieve greater DC and KHN values when tested at simulated intraoral temperatures. A better mold design would be one that simulated the light and thermal properties found in the tooth.

CONCLUSION

Within the limitations of this study, it could be concluded that

- Small differences between room and intraoral temperatures had a significant effect on the DC and KHN of the tested RBCs ($p < 0.05$).
- Cavities with a low volume and a high surface area ratio may need longer light exposure times to ensure an adequate cure at the bottom surfaces.
- Physical properties of the RBC should be tested both immediately and 24 hours after RBC placement, to determine whether there is any increase in the physical properties of the material.

Clinical Relevance

Clinician should look for studies conducted at mouth temperatures, look into reports immediate and 24-hour results. Degree of conversion and KHN values recorded at intraoral temp will be better and the immediate results will be lower.

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