

## EFFECT OF REPEATED PREHEATING AND LIGHT CURING DISTANCE ON FLEXURAL STRENGTH AND DEGREE OF CONVERSION OF TWO RESIN-BASED MATERIALS

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### ABSTRACT

**Objective:** This study was conducted to evaluate the effect of resin-based material, repeated preheating and light curing distance on flexural strength and degree of conversion.

**Materials and Methods:** A total of 220 specimens were used in this study. One-hundred sixty rectangular specimens measuring 12mm x 2 mm x 2mm were dedicated for flexural strength (FS) using three-point bending test. Sixty disc-shaped specimens measuring 4mm x 2mm were dedicated for degree of conversion (DC) using ATR-FTIR spectrophotometer. Specimens were divided into two main groups according to material used; SPRG-containing Beautifil II LS and non-SPRG containing Filtek™ Z250 XT. Each group was subdivided into five subgroups according to the number of preheating cycles to which the syringe was exposed: no preheating, one, five, ten, or fifteen cycles of preheating. Then, each subgroup was further subdivided according to light curing distance (0mm and 10mm). Specimens were stored for 24 hours then subjected to the corresponding testing method (FS n=8, DC n=3). Data was statistically analyzed using three-way ANOVA followed by Tukey's post hoc test.

**Results:** Nanohybrid composite recorded significantly higher FS ( $p<0.001$ ). Also, a significant effect on FS was revealed for preheating ( $p=0.001$ ), light curing distance ( $p=0.038$ ), and the interaction between preheating and distance ( $p=0.027$ ). Meanwhile, giomer recorded significantly higher DC ( $p<0.001$ ). All other variables and their interactions were non-significant.

### Conclusion

1. Material formulation has a significant effect on polymerization efficiency reflected in FS and DC.
2. Preheating and light curing distance had a significant effect on FS, but not on DC.

**Clinical Relevance:** Deeply situated margins require meticulous attention and care from the clinician in order to guarantee seal, adaptation, optimum cure and ultimately restoration success.

**KEYWORDS:** Giomer - Secondary Caries - Polymerization Efficiency - Restoration Longevity - Marginal Seal

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## INTRODUCTION

Despite global calls for preventive dentistry and minimal invasive approaches, multi-surface restorations and deep cavities still exist and are more frequent than patients and clinicians alike expect them to be. Posterior teeth involvement is particularly notable at the occlusal and proximal surfaces<sup>(1)</sup>. Unanimously in such cases, resin composites rank the most reliable direct restorative material with a good survival rate. Nevertheless, resin composites are not without fault.

Direct resin composite annual failure rate has been recognized in the order of 1-3%<sup>(2,3)</sup>. Even with the unceasing advances in composite and adhesive technology, the same findings continue to be reaffirmed where polymerization shrinkage stresses, microleakage, secondary caries and marginal breakdown are what chip away at the clinical longevity of posterior composite restorations<sup>(4,5)</sup>. Even more, *Skudutyte-Rysstad et al.* claimed a composite restoration may serve as a forerunner to lesions in adjacent teeth, as caries incidence in proximal surfaces in immediate contact to composite was more noticeable than in teeth with no adjoining restoration<sup>(6)</sup>.

Whenever a tooth is to be restored, a restorative system is always assessed in the four dimensions of success: mechanical, esthetic, and biological adequacy as well as clinical longevity. Almost always, the primary inclination of clinicians is to judge a material by its mechanical attributes for life expectancy<sup>(7)</sup>. A material is seldom appraised according to its 'biologic return on investment (R.O.I)' or impact on the system to which it is introduced. In essence, the biologic dimension of restoration success universally focuses on pulp vitality and health, gingival and periodontal health, margin seal, tooth restoration and integrity, and re-establishing stable proximal contacts, contours as well as occlusion<sup>(8)</sup>. Inherently, clinical decision making should be rigged in favor of a 'positive

**biologic R.O.I'** where success entails that any intervention/restoration does not cause harm, nor instigates new unnecessary steps or replacement, as it imparts protection and functionality to the tooth, and neighbouring<sup>(9)(10)</sup>.

In view of that, it comes naturally to explore the clinical observations and reasons behind such failures. Generally, areas which harbor biofilm adherence and maturation undisturbed are prone to biological failures. This draws attention to inherent material attributes that may either trigger or suppress biofilm formation relative to that associated with natural sound enamel. In parallel, final restoration surface quality, physiologic contour, and contact tightness are of equal importance<sup>(11)</sup>. According to *Heintze and Rousson*, Class II biological failures in the form of secondary caries or caries adjacent to restoration (CAR) is 8 times more frequent at the gingivo-proximal margin than at the occlusal and axio-proximal margins<sup>(1)</sup>. Gingival margins of class II composites are specifically challenged by difficulty of isolation, matricing, questionable adaptation, and stretches to the insufficiency of light energy reaching the deepest layers of the direct resin-based restoration. Needless to say, gingival seats lacking enamel are more prone to debonding<sup>(11)</sup>. Hence, secondary caries does not occur at margins sealed to perfection, but rather at margins where bond between tooth and restoration have been compromised via uncontrolled polymerization shrinkage stresses or insufficient adaptation. Progressive silent deterioration of material/interface can be expected further with dynamic loading<sup>(12)</sup>, especially when interfacial gap size exceeds 60microns, and when patient caries risk factors align<sup>(13)</sup>. Salvaging the tooth/restoration before catastrophic failure becomes even harder when it goes undetected clinically and radiographically<sup>(14)</sup>.

For that reason, proactive measures upfront to create impervious seal, resistance to demineralization, and superior strength at margins

addresses the prospect for any failure before inception. Nonetheless, up until this moment, no resin composite has zero shrinkage or an absolute 100% cure<sup>(11)</sup>, where cure always falls short within the range of 40-80%<sup>(15)</sup>. This predicament makes composites vulnerable to an oral environment that amplifies shortcomings of any material due to its dynamic and unforgiving nature<sup>(11)</sup><sup>(16)</sup>. It is even further intensified by the unskilled placement of composite, which considerably cuts back at its life expectancy<sup>(17)</sup>. Inadequate polymerization in deeper segments of Class II cavities is a persistent concern. The way a clinician performs curing impinges on polymerization efficiency; with factors like distance and angle of light curing tip relative to restoration surface in addition to thickness, shade, curing time and power. Collectively, these clinical variables affect the actual radiant exposure and light energy received by material and thus the extent to which resin monomer can convert to polymer dictating material properties<sup>(4,15,18)</sup>.

Evidently, for the sake of seal in such a deeply situated area of the proximal cavity, alongside skill and tactile finesse, a reliable material is needed that can guarantee the success of the restoration. In such instances, materials that ensure cure and exhibit maximum flow are the go-to material for their capacity to adapt and self-level. However, flowable materials per se are infamous for their mechanical performance. Also, their polymerization shrinkage is much higher due to the high resin-to-filler ratio<sup>(19)</sup>. According to *Heintze and Rousson*, Class II failures as chipping or bulk fractures are seen more frequently when material flexural strength does not meet the ISO standard of 80MPa for posterior restorations<sup>(1)</sup>. In contrast, conventional composites offer superior mechanical properties, yet have a paste-like consistency that interferes with wetting and adaptation to the geometry of the cavity. This also shows in between increments perceived as 'knit lines'<sup>(16)</sup>. Composites are not condensed but rather gently push-patted in place<sup>(20)</sup>. Still, due to

their relatively high viscosity and the notion of polymerization shrinkage stresses, composites may develop interfacial gaps and microleakage to which the oral environment is not forgiving.

Consequently, choosing one consistency over the other comes at the expense of mechanical performance or instead with zero regard for handling characteristics and subsequent adaptation. Alternatively, introducing preheating or prewarming as an additional preliminary step to resin-based materials suggestively entails low cost, high benefit, and a relatively shallow learning curve. This makes it more approachable by clinicians<sup>(10,21)</sup>. In theory, preheating a conventional, paste-like material creates a temporary state of increased and enhanced molecular mobility that serves as the ideal convergence of handling and endurance prerequisites alike. Briefly, the viscosity of the heavy, highly-filled composite is reduced, making it more yielding, more adaptable and wetting to cavity details, thus decreasing chances for interfacial gap formation. In an ideal scenario, warming composites to pre-set temperatures improves material cure depth, degree of conversion, enhances property development physically and mechanically, even biologically as it decreases biodegradation and cytotoxicity<sup>(22,23)</sup>.

Continuing to optimize current solutions and to explore alternatives to composites seems like the sensible thing to do to find the next-best or a worthy adversary. Any restorative material is expected to adapt or perish according to how well it rises to the challenges of the oral cavity, alongside the clinician's mastery in creating favorable conditions for restoration success. Therefore, this study intends to explore **the clinical conundrum presenting itself; whether resin-based materials can attain optimum properties in deep gingival seats far from the light source**. Altogether, it seems that a material claiming to possess both low-shrinkage and bioactive properties would be ideal for situations where maximum biologic ROI is intended by

mutually restoring the defect, adequately sealing margins, and preserving the health of the approximal tooth surface<sup>(24,25,26)</sup>. In addition, habitual preheating or actively enhancing flow of material seems like a plausible solution to over-ride the pending challenges of sufficiency of cure and adaptation in deep margins of Class II restorations.

### Null Hypothesis:

- There is no difference between the two resin-based materials; SPRG-containing giomer and non-SPRG containing conventional nanohybrid composite with regards to their tested properties; flexural strength and degree of conversion.
- Preheating does not influence the tested properties of the two resin-based materials. Syringes that received no preheating, a single preheating cycle or repeated preheating cycles have no difference.
- Light curing distance (0mm and 10mm) has no influence on the tested properties of the two resin-based materials.

### AIM OF THE STUDY

This study was conducted to evaluate the photopolymerization efficiency in deeply-situated margins with regards to the following:

- Primary outcome: the effect of resin-based material type (one SPRG-containing giomer and one non-SPRG-containing), preheating and light curing distance
- Secondary outcomes:
  - Flexural Strength
  - Degree of Conversion

### MATERIALS AND METHODS

Research proposal was presented to and approved by the Research Ethics Committee at Faculty of Dentistry Ain Shams University, Cairo,

Egypt (FDASU-Rec ER052340). Included, a power analysis was designed to have adequate power to apply a statistical test of the null hypothesis that there is no difference between the different groups regarding flexural strength and degree of conversion. By adopting an alpha ( $\alpha$ ) and beta ( $\beta$ ) levels of 0.05 (5%) (i.e., power = 95%), and effect sizes (f) of 1.10 for flexural strength and (f) of 2.58 for degree of conversion; calculated based on the results of a previous study<sup>(27)</sup>; the minimal required total sample size was found to be 60 samples for flexural strength; FS (3 per group) and 40 samples for degree of conversion; DC (2 per group). Specimens were increased to 8 per group and 3 per group, respectively. Sample size calculation was performed using G\*Power version 3.1.9.7.<sup>(28)</sup>

### Study Design:

A total of 160 specimens for flexural strength test (FS) and 60 specimens for degree of conversion (DC) were divided into 20 groups (n = 8 for FS, n = 3 for DC) according to the three levels of the study; Level 1: the resin-based material used (either *SPRG-containing giomer* or *non-SPRG-containing nano-hybrid composite*), Level 2: the number of preheating cycles (0, 1, 5, 10 or 15 cycles) , and Level 3: the light curing or irradiation distance (0mm or 10mm distance). *Levels of the study and experimental groups, for each of the tests performed are shown in Figure 1.*

In this study, two types of resin-based materials were investigated (**Table 1**):

1. One SPRG-containing resin-based material (**Beautiful II LS Giomer**, SHOFU INC., Kyoto, Japan.)
2. One non-SPRG-containing resin-based material (**Filtek™ Z250 XT Nano-hybrid Composite**, 3M ESPE DENTAL PRODUCTS St. Paul, MN, USA.)

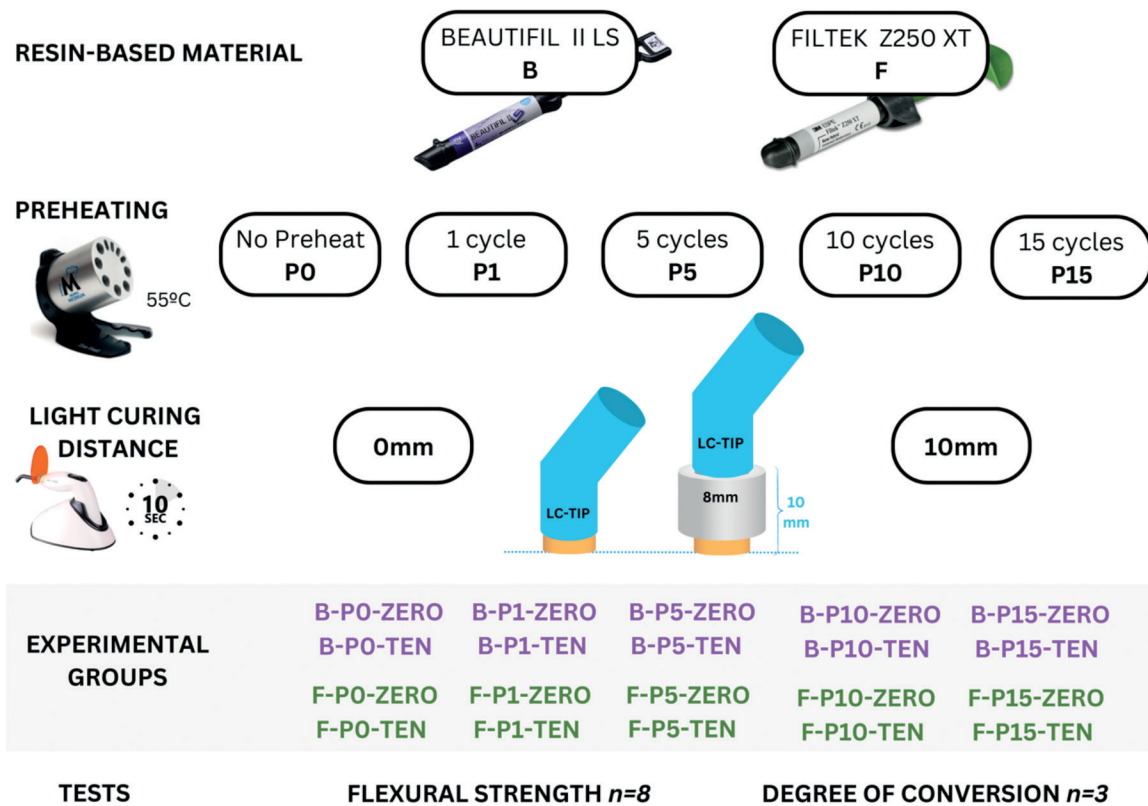


Fig. (1) Levels of the Study and Experimental Groups.

TABLE (1) Materials used in this study.

Material	Composition	Manufacturer & Batch No.
<b>Beautiful II LS</b> Polymer-based, Light-cured, dental Restorative Material with GIOMER technology (Radiopaque), 4g Shade A2	<b>Organic Matrix:</b> Urethane diacrylate, Bis-MPEPP, Bis-GMA, TEGDMA, Polymerization initiator, Pigments and others <b>Filler Content:</b> S-PRG filler (fluoro- boroaluminosilicate glass) <b>Filler % (wt):</b> 82.9% by weight (68.6% by volume) with a particle size of 0.01-4, mean 0.8 <sup>(29)</sup>	<b>SHOFU INC.</b> , Kyoto, JAPAN  <b>LOT 032263 – REF PNY2271</b>
<b>Filtek™ Z250XT</b> Nanohybrid Universal Polymer-Based Dental Restorative Material, 3g Shade A2	<b>Organic Matrix:</b> Bis-GMA, UDMA, Bis-EMA, PEGDMA, and TEGDMA Resins <b>Filler Content:</b> Combination of surface modified zirconia/silica and 20nm surface modified silica particles. <b>Filler % (wt):</b> 81.8% by weight (67.8% by volume) with a particle size of 20nm for silica and approximately 0.1-10 microns for the zirconia/silica.	<b>3M ESPE DENTAL PRODUCTS</b> , ST PAUL, MN, USA <b>LOT NE57136– REF 1470A2</b>

Abbreviations: **Bis-MPEPP:** 2,2-bis(4-methacryloxyphenyl) propane; **Bis-GMA:** bisphenol A diglycidyl methacrylate; **TEGDMA:** triethylene glycol dimethacrylate; **S-PRG:** surface pre-reacted glass; **UDMA:** diurethane dimethacrylate; **Bis-EMA:** bisphenol A diglycidyl methacrylate ethoxylated; **PEGDMA:** poly-ethylene glycol dimethacrylate.

**Preheating:**

For the purpose of this study, a standard warmer or heater was used (ENA Heat, Composite Heating Conditioner CHC3, GRUPPO MICERIUM®, Italy; S.N. 2022-CO819). It was adjusted to its second temperature settings at 55°C. Vacant heater was allowed the full 55-60 minutes to attain the anticipated temperature as instructed by the manufacturer in device manual. Afterwards, and according to experimental design, a non-refrigerated syringe was docked in heater. Preheating time was standardized throughout procedures at a full 20 minutes to account for syringe bulk/full volume and to be clinically reasonable.<sup>(30)</sup>

To explore the hypothetical effect of repeated preheating, multiple preheating and cooling cycles were performed measured up against a *no preheating group* (P0), where syringe received no preheating whatsoever and was used at room temperature (non-refrigerated)<sup>(31)</sup>. The maximum number of cycles was determined by an initial hands-on tryout. A Typodont tooth was prepared yielding a medium-sized class II cavity with deep proximal margin to reflect the study's interest to highlight the effects of light distance and attenuation on deep margin management with preheated direct resin restoration<sup>(32)</sup>. This consumed 0.25g; a compute equivalent. Accordingly, an assumption was calculated that a single 3-4g syringe can suffice for approximately 12-16 medium-sized cavities with deep proximal margins. Fittingly, 15 cycles was chosen as the maximum to account for any discrepancy or waste. Then, to make room to observe potential effects of *repeated preheating*, measurements were made along spaced out intervals or increments of 5 cycles up until finishing a single syringe<sup>(33)</sup>. Therefore, final experimental design compared no preheating, one, five, ten and fifteen cycles of preheating.

To perform preheating, for Group P1, a single preheating cycle consisted of 20 minutes at

55°C was performed, after which material was *immediately* inserted into designated mold. This is in accordance to literature, where it is imperative to shorten the total time between the actual heating of the material, opening the syringe, acquiring an increment, and placing into the cavity for curing. Owing to the relatively small-scale dimensions of the test specimens, keeping total time of **transfer-pack-and-cure** to a minimum was feasible against a digital timer. For groups P5, P10 and P15, undergoing multiple preheating, a full cycle consisted of a single preheating cycle of 20 minutes followed by cooling or return to room temperature (outside of heater) for 15 minutes<sup>(16)</sup>. Afterwards to commence another full cycle, syringe was returned into heater and so on. This was iterated for the number of times as dictated by the experimental design (namely five, ten and fifteen). Care was exercised to use full syringes within same session, without waste or transferring a syringe to another day/week to control preheating-cooling cycles and time given to material to recover across this study.

**Flexural Strength:**

Specimens were prepared as described by *Yap and Teoh*, using a specially-constructed split copper mold with a rectangular central hollow space measuring 12mm in length, 2mm in width and 2mm in thickness (Figure 2a). The split mold was assembled and placed over a celluloid strip (Transparent Strips, TOR VM Ltd, Moscow, Russia) over a glass slab. The resin material was packed into the mold with a burnisher and a celluloid strip was then placed onto the surface of the resin-based material. Gentle pressure was applied using a thin glass slide to confirm compactness and extrude any excess material<sup>(34)</sup>. The mean time between transferring composite from the heating unit, packing into mold and cure initiation was 40 seconds for all tests<sup>(33,35,36)</sup>.

Curing was performed using a light curing unit (Led-F High-Intensity Curing Light, Guilin

Woodpecker Medical Instrument CO, LTD, Guangxi, China; S.N. L1900731F) with output intensity of  $1600 \text{ mW/cm}^2$  and light cure tip diameter of 8mm. The intensity of the curing unit was periodically checked using a radiometer (CM300-2000, Curing Light Meter, Apoza). Curing was performed according to manufacturer instructions for 2mm thickness in *either* materials as 10 seconds. To cure the 12-mm mini-flexural specimens end to end, an overlap method was adopted. Light curing tip was positioned intimately onto the top celluloid strip; first in the middle of the specimen, then at both sides of the specimen (Figure 2b). No supplementary curing was performed for the specimen from the bottom surface to restrict irradiance to clinical reality<sup>(16, 34, 37)</sup>.

For 0mm distance, the specimen was light cured directly over the celluloid strip after removal of

the glass slide as described. In contrast, specimens tested at 10mm distance required using a specially-constructed spacer (Figure 2c) in the form of a rectangular stainless steel piece with a central opening of 6mm-width and 25mm-length to ensure stable positioning of the 8mm-diameter light curing tip. The 10mm light curing distance was determined by the distance from the bottom of the specimen to the light curing tip, which equates to the 2mm thickness of the specimen plus the 8mm thickness of the metal piece or spacer, (Figure 2d).

Before disassembling the mold, top surface of specimen was marked for identification at testing using a standard Sharpie permanent marker. Specimens were finished gently using #600 SiC abrasive papers from both long sides to remove any flashes, while top and bottom surfaces were left unaltered. Using a digital caliper (Mitutoyo

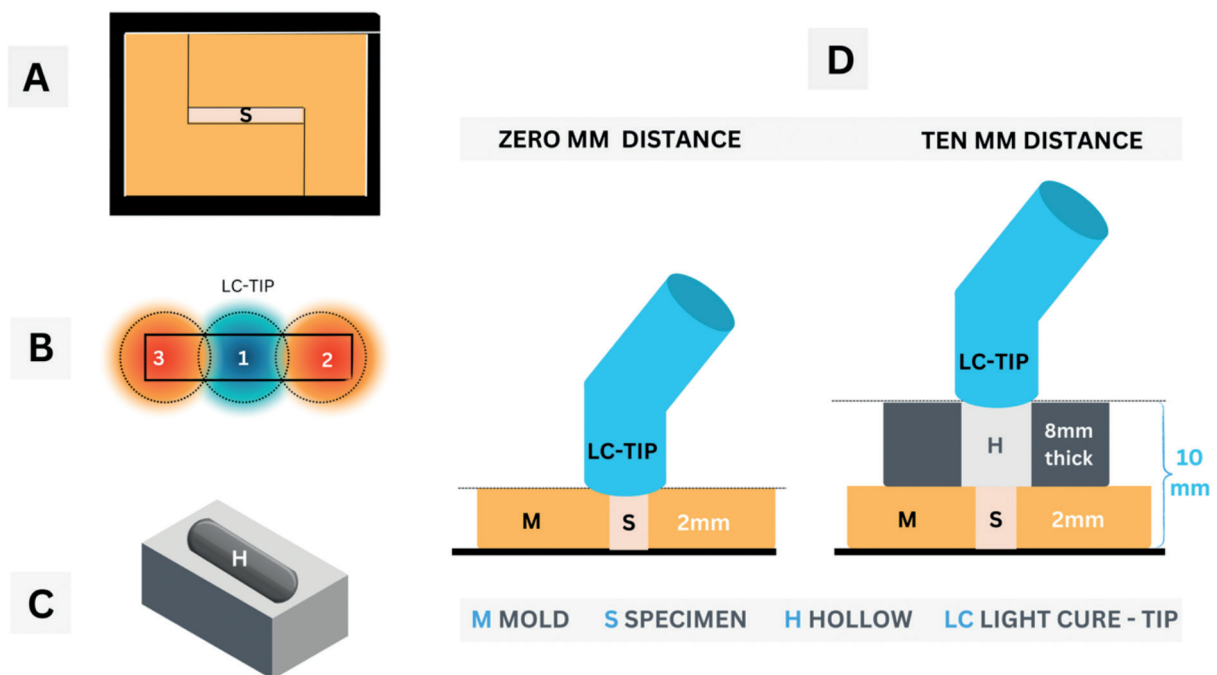


Fig. (2) Schematic representation of a) Custom mold for FS, b) Overlap curing method, c) Spacer used for light curing distance, d) Curing assembly for FS at 0 and 10mm distance.

ABSOLUTE Digimatic Caliper Series 500-196, Mitutoyo Corp, USA; S.N. 0353916) specimens were measured in length, followed by width and thickness in two different locations along the specimen. Measurements were averaged to calculate the length, average width and average thickness for each specimen.

All specimens were then stored dry in individual labeled Eppendorf 1.5mL clear microcentrifuge tubes (Eppendorf Tubes® 3810X, Hamburg, Germany), at room temperature for 24 hours. Specimens were kept in a light-proof container and additionally wrapped in aluminum foil to block any passageway for ambient light<sup>(27,36)</sup>.

After 24 hours, the prepared specimens were subjected to three-point bending test in a Universal Testing Machine (LR5K series, Lloyd Instruments, Ltd, UK), operated using Nexygen Software Version 4.6 at the Biomaterials Testing Unit, Faculty of Dentistry, Ain Shams University. Each specimen was positioned, supported by two mounted parallel rods (2mm in diameter), with a 10mm distance between their centers. The load was applied at the center of each specimen up to failure through a load applicator ending with a third rod (2mm in diameter). The test was run at a crosshead speed of 0.5mm/min. The flexural strength was calculated according to the equation:

$$\text{Flexural Strength} = 3FL/2BH^2,$$

where F is the maximum load in Newton (N), L is the distance between supports in mm; B and H are the specimen width and height respectively in mm. Values were expressed in MegaPascal (MPa)<sup>(34)</sup>.

### Degree of Conversion:

A specially-constructed copper mold with central circular opening 4mm in diameter and 2mm in thickness was used to prepare disc-shaped specimens (Figure 3a). The mold was placed over a glass slab with an intervening celluloid strip.

Resin material was inserted into the assembled mold and another celluloid strip was placed onto the surface of composite covered with a glass slide. Then, gentle pressure with slide was applied to extrude any excess material. The resin materials were light cured for a single 10 seconds according to manufacturer instructions from top surface only. The mean time for material **transfer-pack-and-cure** was synchronized at 40 seconds as previous test<sup>(35,36)</sup>.

For 0mm distance, the specimen was light cured directly over the celluloid strip after removal of the glass slide. For specimens light cured at 10mm distance, the 8mm-diameter light tip was rested on cylindrical copper ring with a central hole of 6mm diameter (Figure 3b). The 10mm light curing distance was determined as for flexural strength specimens, equal to the 2mm thickness of the specimen plus the length of the internal hollow of 8mm (Figure 3c).

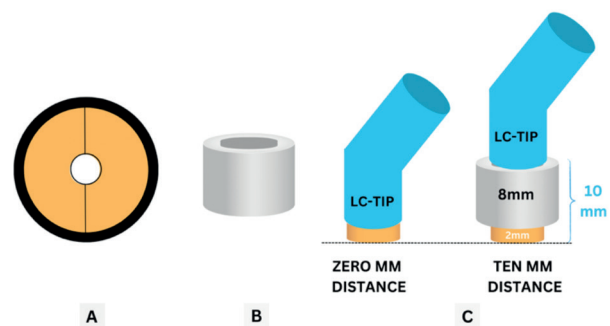


Fig. (3) Schematic representation of a) Custom mold for fabricating DC specimens, b) Spacer used for light distance, c) Curing assembly at 0mm and 10mm distance for DC.

Molds were disassembled and top surfaces of specimens were marked from the side of each specimen near its top surface using Sharpie permanent marker. Flashes were carefully removed using Bard Parker blade #11 (Xinda Surgical Blades, Wuxi Xinda Medical Device Co., Ltd.). Specimens



were kept in dry storage, in a light-proof container for 24 hours at room temperature in a manner similar to flexural specimens<sup>(38,39)</sup>.

### Degree of Conversion Testing: (ATR-FTIR Spectroscopic Measurement)

After 24 hours of being stored dry at room temperature in a light-proof container, specimens were subjected to degree of conversion testing<sup>(38)</sup>. DC was assessed with an FTIR spectrometer (Bruker ALPHA II, Compact FTIR Spectrometer, Bruker Optik GmbH, S.N. 12573756) with an attenuated total reflectance accessory (Bruker Optics Platinum ATR high-performance, QuickSnap™ accessory, diamond crystal). ATR-FTIR was connected to an external computer running on the OPUS software Version 7.8. Test was performed at the Central Lab Unit, Faculty of Science, Ain Shams University.

To initiate the experiment, first the ATR monolithic diamond crystal was cleaned with alcohol then left to dry. Background spectra of air was collected, followed by spectra for specimens. Each specimen was carefully positioned over the crystal. The mark at the side of each specimen was reviewed for differentiation of top and bottom surfaces. After which, an anvil was pressed down to ensure close contact with the specimen surface being tested. Both top and bottom surfaces were analyzed. FTIR absorbance spectra ranging from 400 to 4000  $\text{cm}^{-1}$  were documented by 32 scans at a resolution of 4  $\text{cm}^{-1}$ . Independently, the FTIR absorbance spectra of the two uncured resin-based materials were recorded as well<sup>(40) (41) (42)</sup>.

Afterwards, degree of conversion expressed as percentage (DC %) per surface was calculated relying on the following equation<sup>(16) (39)</sup>:

$$\text{DC (\%)} = 1 - [\text{C/U}] \times 100$$

where: C = aliphatic C=C /aromatic C=C of polymer, and

U = aliphatic C=C /aromatic C=C of monomer

The equation calculates the changes in peak intensities of aliphatic and aromatic carbon double bonds C=C between the cured polymer (C) and uncured monomer (U) states of the material. For all Beautifil II LS specimens, the internal reference peaks were consistent for aliphatic and aromatic as 1638 $^{-1}$  and 1598 $^{-1}$  respectively. For all Filtek Z250 XT, the internal reference peaks were consistent for aliphatic and aromatic as 1635 $^{-1}$  and 1608 $^{-1}$  respectively<sup>(16)</sup>.

## RESULTS

Data was collected, tabulated and statistically analyzed.

### Statistical analysis:

Numerical data was represented as mean and standard deviation (SD) values. Shapiro-Wilk's test was used to test for normality. Homogeneity of variances was tested using Levene's test. Data showed parametric distribution and variance homogeneity and were analyzed using three-way ANOVA followed by Tukey's post hoc test. Comparisons of simple main effects was done utilizing the error term of the three-way model with p-values adjustment using Bonferroni correction. The significance level was set at  $p < 0.05$  within all tests. Statistical analysis was performed with R statistical analysis software version 4.3.1 for Windows<sup>(43)</sup>.

### Flexural Strength:

Results of three-way ANOVA showed that there was a significant effect of material ( $p < 0.001$ ), preheating ( $p = 0.001$ ) as well as light curing distance ( $p = 0.038$ ) on flexural strength. Results also showed that there was a significant interaction between preheating and light curing distance ( $p = 0.027$ ). Intergroup comparisons showed that non-SPRG containing, nanohybrid composite, Filtek Z250 XT consistently displayed higher flexural strength values compared to SPRG-containing, giomer

Beautiful II LS; at all preheating cycles and light curing distances ( $p < 0.001$ ), whereas significance for P15-0mm distance ( $p = 0.010$ ). Within P0, Beautiful II LS samples cured at 10 mm had significantly higher value than samples cured at 0 mm ( $p < 0.001$ ). Within P10 and P15, samples cured at 0 mm had significantly higher values than samples cured at 10 mm ( $p = 0.002$  and  $p = 0.021$ , respectively). Other comparisons (P1 and P5) were not statistically significant ( $p > 0.05$ ). For Filtek Z250 XT, no statistical significant difference was recorded between all groups ( $p > 0.05$ ) at all conditions.

Results of intergroup comparisons presented in Table (2) showed that for Beautiful II LS cured at 10 mm, there was a significant effect of number of preheating cycles on flexural strength ( $p < 0.001$ ). Post hoc pairwise comparisons ranked P0 (no preheating) as significantly highest flexural strength, followed by P1 and P5, then P10 ( $p < 0.001$ ). All

other comparisons were not statistically significant ( $p > 0.05$ ).

**Degree of Conversion (Top):**

Results of three-way ANOVA showed that only type of material had a significant effect on degree of conversion for top surface ( $p < 0.001$ ). Meanwhile, number of preheating cycles ( $p = 0.621$ ) and light curing distance ( $p = 0.986$ ) showed no significant effect on degree of conversion of top surfaces of the 2mm-thick specimens, as well as on bottom-to-top ratio ( $p > 0.05$ ). Intergroup comparisons showed that Beautiful II LS consistently presented significantly higher DC values than Filtek Z250 XT ( $p < 0.05$ ), except for P0 specimens cured at 10 mm where both materials showed no difference. Meanwhile, regardless of resin-based material and preheating, there was no significant difference between both light curing distances ( $p > 0.05$ ).

TABLE (2) Effect of preheating on flexural strength, expressed in (MPa).

Material	Distance	Flexural Strength (MPa) (Mean±SD)					f-value	p-value
		P0	P1	P5	P10	P15		
Beautiful II LS	0 mm	94.12±7.38 <sup>A</sup>	104.54±8.83 <sup>A</sup>	97.35±4.31 <sup>A</sup>	105.00±8.00 <sup>A</sup>	102.08±9.44 <sup>A</sup>	2.94	0.051
Beautiful II LS	10 mm	109.46±6.76 <sup>A</sup>	100.56±3.97 <sup>B</sup>	99.54±5.96 <sup>B</sup>	90.18±7.79 <sup>C</sup>	92.32±4.82 <sup>BC</sup>	12.86	<0.001*
Filtek Z250 XT	0 mm	147.12±17.56 <sup>A</sup>	146.92±15.71 <sup>A</sup>	142.85±21.10 <sup>A</sup>	137.89±13.14 <sup>A</sup>	124.74±19.49 <sup>A</sup>	2.22	0.087
Filtek Z250 XT	10 mm	141.55±13.39 <sup>A</sup>	132.45±16.36 <sup>A</sup>	141.78±14.47 <sup>A</sup>	126.49±11.67 <sup>A</sup>	127.89±12.08 <sup>A</sup>	2.28	0.080

Means with different superscript letters within the same horizontal row are significantly different; \*significant ( $p < 0.05$ ).

TABLE (3) Effect of preheating on degree of conversion (top), expressed as (%).

Material	Distance	Degree of Conversion (Top) (%) (Mean±SD)					f-value	p-value
		P0	P1	P5	P10	P15		
Beautiful II LS	0 mm	61.72±1.39 <sup>A</sup>	61.63±1.51 <sup>A</sup>	61.31±0.63 <sup>A</sup>	61.97±1.08 <sup>A</sup>	60.80±1.11 <sup>A</sup>	0.50	0.735
Beautiful II LS	10 mm	60.26±2.93 <sup>A</sup>	62.85±1.04 <sup>A</sup>	61.73±0.53 <sup>A</sup>	61.52±1.08 <sup>A</sup>	61.75±1.07 <sup>A</sup>	1.04	0.433
Filtek Z250 XT	0 mm	58.59±0.50 <sup>A</sup>	57.79±0.14 <sup>AB</sup>	57.43±0.21 <sup>B</sup>	57.40±0.30 <sup>B</sup>	57.05±0.40 <sup>B</sup>	9.06	0.002*
Filtek Z250 XT	10 mm	57.99±0.78 <sup>A</sup>	57.10±0.68 <sup>A</sup>	57.46±0.68 <sup>A</sup>	57.75±0.62 <sup>A</sup>	57.22±0.16 <sup>A</sup>	1.05	0.427

Means with different superscript letters within the same horizontal row are significantly different; \*significant ( $p < 0.05$ ).

Results of intergroup comparisons presented in Table (3) showed that all comparisons between groups of Beautifil II LS at 0 mm and 10 mm light curing distance, as well as Filtek Z250 XT at 10 mm distance showed no statistical significant difference ( $p>0.05$ ). In contrast, Filtek Z250 XT cured at 0 mm, revealed significant difference between different preheating cycles ( $p=0.002$ ). Post hoc pairwise comparisons showed that F-P0-Zero and F-P1-Zero recorded significantly higher DC value than groups receiving five, ten and fifteen preheating cycles ( $p<0.001$ ).

#### Degree of Conversion (Bottom):

Results of three-way ANOVA showed that only type of material had a significant effect on DC of bottom surfaces of 2mm-thick specimens

( $p<0.001$ ), while preheating ( $p=0.472$ ) and light curing distance ( $p=0.313$ ) had no significant effect. Intergroup comparisons showed that again Beautifil II LS presented significantly higher DC values than Filtek Z250 XT ( $p<0.05$ ) at P0-0mm, P1 and P15 both distances, whereas materials showed no difference at P5 and P10 cured at both distances. In tandem, specimens of Filtek Z250 XT preheated once and Beautifil II LS preheated 15 times, cured at 0 mm distance (F-P1-0mm, B-P15-0mm) had significantly higher values than those cured at 10mm distance ( $p<0.05$ ). Other comparisons were not statistically significant ( $p>0.05$ ). Finally, results of intergroup comparisons presented in Table (4) showed that regardless of material and curing distance, the effect of number of preheating cycles was not statistically significant ( $p>0.05$ ).

TABLE (4) Effect of preheating on degree of conversion (bottom), expressed as (%).

Material	Distance	Degree of Conversion (Bottom) (%) (Mean±SD)					f-value	p-value
		P0	P1	P5	P10	P15		
Beautifil II LS	0 mm	61.49±0.97 <sup>A</sup>	61.02±1.17 <sup>A</sup>	64.51±6.83 <sup>A</sup>	59.94±3.43 <sup>A</sup>	61.99±0.13 <sup>A</sup>	<b>0.67</b>	<b>0.627</b>
	10 mm	59.28±3.10 <sup>A</sup>	61.04±0.51 <sup>A</sup>	59.20±0.97 <sup>A</sup>	60.46±3.56 <sup>A</sup>	60.29±0.20 <sup>A</sup>	<b>0.41</b>	<b>0.801</b>
Filtek Z250 XT	0 mm	57.17±1.24 <sup>A</sup>	56.52±0.39 <sup>A</sup>	56.08±0.62 <sup>A</sup>	57.10±0.70 <sup>A</sup>	56.18±0.53 <sup>A</sup>	<b>1.36</b>	<b>0.315</b>
	10 mm	60.44±5.90 <sup>A</sup>	54.44±0.93 <sup>A</sup>	58.79±4.28 <sup>A</sup>	56.45±2.38 <sup>A</sup>	54.98±1.90 <sup>A</sup>	<b>1.54</b>	<b>0.263</b>

Means with different superscript letters within the same horizontal row are significantly different; \*significant ( $p<0.05$ ).

## DISCUSSION

Restoration integrity at deeply-situated, cervical, proximal margins will always be a key player in the permanence of resinous restorations. Intuitively, seal at margins is non-negotiable, alongside optimization of cure in that area. In essence, techniques as deep margin elevation/management emerged to enable clinicians to best handle the challenging margin position and state utilizing a diverse array of

materials, formulations, and leveraging the benefits of thermal energy or thixotropy<sup>(44,45)</sup>.

Preheating has been used by many practitioners with composites, glass ionomers, even resin cements, all in the name of improving material-tooth association as well as physical and mechanical properties<sup>(46)</sup>. As with all trends ushering into clinical dentistry with good intention, these in-house, smart solutions may do more harm than good. Recently,

much focus has gone to bulk-fill materials for their aptitude to save time in restoring large cavities<sup>(16)</sup>. Still, the need to look into the effect of preheating and specifically repeated preheating is imperative as clinicians may inquisitively experiment with materials readily available in practice. Although the effect of preheating on viscosity and other properties was found to be generally favorable, it is sometimes inconsistent and material-dependent<sup>(21)</sup>. For that reason, evidence-based practices and recommendations that make a distinction between different resin-based material formulations and takes into account coexistent clinical challenges are indispensable. Thus far, no study explicitly investigated the potential counter-effect of preheating on assumed insufficient cure in far, deep proximal margins. Attempts were only made to override the need for more cure time by preheating the material<sup>(46-49)</sup>.

Research investigated several preheating approaches from using water baths, hot ovens, incubators, commercial composite warmers housing compules and syringes alike, and even a more progressive gun-style warmer that can jointly heat and transfer material into cavities. Reassuringly, heating devices including AdDent Calset Warmer, BioClear™ HeatSync and ENA Heat were found to perform consistently with manufacturer claims in regards to temperatures reached and time required for heating<sup>(16) (46) (47) (48) (49)</sup>. Likewise, in this study, a standard heater (ENA Heat) set at temperature 55°C was elected for uniform heating well above room temperature, equally tolerable by pulp as 68°C, but without the risk of cellular toxicity<sup>(50)</sup>.

This also takes into account that maximizing benefit from preheating can only occur within a very specific window of time where curing capitalizes on the captured warmth, otherwise it becomes obsolete regardless the amount of thermal energy induced<sup>(21)</sup>. Resins heated even as high up to 68°C plunged fast by 50% at the 2-minute mark and by 90% at the 5-minute mark<sup>(16)</sup>. Hence, best practices recommend to skillfully work around

this foreseen drop in temperature by immediate transfer of material to cavity, sculpting and prompt cure. Considering it is expected in syringes must faster than in compules, this study adopted the least documented time of 40 seconds as the total *transfer-pack-and-cure* time. Also, total time of heating performed was 20 minutes keeping it within reasonable range applicable in clinical setting<sup>(30)</sup>. In the same way, use of non-refrigerated syringes was intentional to avoid any thwarting of DC as proven by *El-Maksoud et al*<sup>(31)</sup>.

According to the results of this study, the null hypothesis could be partially rejected. Resin-based material as a parameter had a significant effect on flexural strength ( $p < 0.001$ ), as well as on degree of conversion for both top and bottom surfaces ( $p < 0.001$ ), with no revealed effect on DC ratio (bottom to top,  $p > 0.05$ ). Filtek Z250 XT recorded higher flexural strength values compared to Beautifil II LS. This may be attributed to the fundamental understanding that strength of 'any composite structure' is a function of filler type and loading. Once again, a wide distribution of particle size proved best to facilitate higher loading and higher FS in favor of Filtek Z250 XT. So, although SPRG-containing Beautifil II LS performed within the clinical acceptable range, it did not prove itself an extraordinary alternative to non-SPRG nanohybrid composite mechanically in these testing conditions. On the other hand, *Kimyai et al.* registered higher flexural strength values for giomer with a single 15-minute preheating cycle, albeit against a microhybrid composite. They attributed their results to enhanced degree of conversion without actually testing DC<sup>(30)</sup>.

On the other hand, in this study, it seems that the specific resin mixtures of the low-shrinkage Beautifil II LS promoted higher degrees of conversion; leaning towards lower initial viscosity, higher molecular flexibility, plus less interfacial scatteration of light<sup>(40,51)</sup>. DC values may also be a reflection of the interaction of photoinitiator

efficiency, curing time and intensity relative to limited increment thickness<sup>(52)</sup>.

Preheating can potentially act as a driver to polymerization reaction kinetics generally enhancing the amount of monomer converting to polymer<sup>(18,46)</sup>. However, no clarity existed on the effect of multiple iterations of preheating on resin-based materials. The results of this study show that preheating cycles had a significant effect on flexural strength ( $p=0.001$ ), with no effect on top and bottom degree of conversion ( $p>0.05$ ). This is in agreement with the works *Ribeiro et al.*<sup>(16)</sup>. In this study, FS values for Beautifil II LS cured at 0mm revealed no significant effect of preheating or repeating preheating ( $p>0.05$ ). However, for giomer specimens cured at 10mm, there was a significant effect of number of preheating cycles on FS ( $p<0.001$ ), where highest FS was recorded by no preheating group. This was followed by groups P1 and P5. Such a finding may reveal a threshold number of preheating after which giomer may demonstrate altered polymerization behavior. This may demand the switch to single-use compules in case of preheating<sup>(36)</sup>. In contrast, nanohybrid composite demonstrated consistent behavior, at all preheating and repeated preheating conditions, when cured at both 0mm and 10mm distances ( $p>0.05$ ). Furthermore, Filtek Z250 XT showed higher top surface DC values by no preheating. This disparity with other studies may be on account of varying methodology where composite syringes were placed in warmers for times varying from 3 minutes to 15 minutes,<sup>(47)</sup> with some going over and beyond to 30-40 minutes per cycle or multiple days<sup>(41)</sup>, or even for 40 cycles<sup>(33)</sup> <sup>(36)</sup>, besides the difference in material thickness. In addition, times where composite syringe was allowed to recover back to room temperature was vague and undeclared in some studies.

Given thickness is limited to 2mm, both materials only require 10 seconds exposure time at 1000 mW/cm<sup>2</sup>. However, 12mm x 2mm x 2mm FS specimens were cured at 1600 mW/cm<sup>2</sup> using

an overlap method to make up for the discrepancy in size between light cure diameter tip (8mm) and full, standard-testing specimen length. In contrast, DC specimens measuring 4mm in diameter were fully covered by the circumference of the light cure tip. Consequently, DC specimens received a 'true', single, 10-second exposure time across the 2mm thickness. Altogether, the total radiant exposure or amount of energy received by the materials was different for both tests<sup>(15)</sup>.

In retrospect, performing DC testing on the exact same specimens of FS may have steered results differently. In the alternative scenario, fractured FS specimens are scrapped, ground with potassium bromide then pressed into a disc/pellet for FTIR. This saves a lot of material consumed in the fabrication of independent DC specimens. More importantly, it allows for the correlation of results of both FS and DC more efficiently per specimen via identical cure conditions. As observed with *Ribeiro et al.*, authors had a better chance explaining material behavior depicted in FS, elastic modulus, DC, and Knoop hardness results even when using longer 20 second exposure time with the 4mm-thick bulk-fill materials. All the same, *Ribeiro et al.* suspected the overlap curing method concealed differences between preheating methods and temperatures tested cured at zero distance<sup>(16)</sup>. From a mechanical perspective, flexural strength is a huge predictor to a restoration's clinical performance. Hence, an alternate method to 'overlap cure' needs to be considered in all future tests, including further modifying dimensions to 8mm x 2mm x 2mm as performed by *Oh et al*<sup>(27)</sup>.

Finally, this may explain the conflicting verdicts on distance where *Oh et al* found irradiation distance to be extremely detrimental to FS and DC<sup>(27)</sup>. In this study, it was expected for distance to have an effect on radiant exposure, by dissipation of energy subtracted from the total emittance from the light cure tip<sup>(15)</sup>, as demonstrated by *Rode et al.*<sup>(53)</sup>. Previous analyses went to great lengths

comparing 0mm to 2mm, 4mm, 6mm, 8mm and up to 10mm. Many used an incremental increase in distance farther away from specimen bottom, while other studies chose to measure up cure at 0mm against the extreme of 10mm as preferred within this study. Either way, some studies demonstrated a significant effect<sup>(54-57)</sup>, while others not<sup>(58)</sup>. In this study, light curing distance had a significant effect only on FS ( $p=0.038$ ), with no effect on top and bottom DC ( $p>0.05$ ). Similarly, all interactions had no statistical significant effect on properties tested ( $p>0.05$ ), except for interaction of preheating and light curing distance on flexural strength ( $p=0.027$ ). Furthermore, it may be that Beautifil's FS at 0mm and 10mm did not mirror Filtek's consistency on account of a difference in cure rate and network densification as induced by preheating yet tempered by distance<sup>(59-62)</sup>. In addition, increment thickness (larger than 2mm) may be better seasoned to investigate such interactions<sup>(63)</sup> with a closer look at depth dependence.

In conclusion, holding clinicians accountable for restoration serviceability has raised the bar in practice to take into consideration all relevant patient, operator and material factors influencing the process<sup>(11-17)</sup>. Ensuring cure at all costs in deeply situated areas is a necessity<sup>(64)</sup>. Even when attempting high degree of conversion for mechanical properties, it is important to remember that according to Watts': 'optimum DC and minimal shrinkage are antagonistic goals' which may in turn jeopardize adaptation<sup>(65)</sup>. Research must continue to go hand in hand to validate and recommend best practices in challenging clinical settings.

## CONCLUSIONS

*Within the limitations of this study,*

1. Material formulation has a significant effect on polymerization efficiency reflected in FS and DC.
2. Preheating and light curing distance had a significant effect on FS, but not on DC.

## Limitations

This study explored exclusively two materials, only permissible in an increment thickness of 2mm. Testing 4mm increment thickness as tolerable by bulk-fill materials may give further room to demonstrate: a) energy dispersion along distance in air, b) compounded by light curing energy attenuation with increasing material thickness c) at varying depths. Then and there, the interaction of the dual variables of light distance with preheating may cause materials to behave differently with regards to their polymerization kinetics. Another parallel direction for research is to investigate the effect of preheating on consequent material polymerization shrinkage stresses and the actual observed impact on adaptation to tooth substrate.

## Conflict of Interest Declaration

The author declares having no competing proprietary, financial or personal interests of any nature in any product, service, and/or company that appeared within the work presented in this article.

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