

Comparative Evaluation Of The Effects Of Preheating On The Microhardness Of Commercially Available Bulk Fill Composite Resins And Conventional Composite Resin -An In-Vitro Study

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ABSTRACT

Background. Composite resin contributes to multifarious success of modern biomaterial research. However, to overcome the disadvantages of the conventional composites, bulk-fill composite resins have been introduced. Those may be inserted in the dental cavity in increments of 4 mm up to 5 mm thick. Current research is focused on the microhardness of preheated commercially available bulkfill composite resins in an In-vitro context.

Aim. To investigate the effect of preheating three bulkfill composite resin and one conventional composite resin on the microhardness of these composites and in turn to uncover the beneficial potential to contribute to the field of dental material science.

Materials and Methods. Three commercial bulkfill resin based composite materials such as Ivoclar Tetric N-Ceram bulkfill, 3M Filtek bulkfill, Tokuyama Estelite alpha and one conventional composite resin-Dentsply Sirona Neospectra ST Universal composite are evaluated in this study. 6 samples from each group are produced using Teflon moulds measuring 4mm & 2mm thickness and 8mm in diameter. Preheated the materials using composite warmer. Vickers hardness number (VHN) values are measured immediately after preheating the samples, and after being stored for 24 hours in distilled water at room temperature. The values on the top and bottom surfaces are calculated.

Results. The Vickers hardness number (VHN) varied according to the composite material, preheating condition, specimen thickness, surface evaluated, and storage period. Based on the numerical values presented in Tables 3 and 4, 3M Filtek Bulk Fill demonstrated comparatively higher VHN values, whereas Tokuyama Estelite Alpha generally demonstrated lower values. Changes between baseline and 24-hour measurements varied according to the material and experimental condition. alpha bulkfill while preheating and on 2mm and 4mm application.

Conclusion. According to the results of this invitro study, the microhardness of the materials differs according to the composition. All tested materials reached a 2mm depth of cure. Preheating increased the microhardness of all the bulkfill composites.

Keywords: Bulkfill Composites, Microhardness, Preheating, Vickers Hardness Number, Depth of cure.

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INTRODUCTION

Resin-based dental composites are promising tooth-resembling materials in restorative dentistry [1]. Its esthetic appearance has been appreciated over the recent years. The patient demand for aesthetic restorations has led to the development of new composite resin technologies.

Recently bulk fill composites were introduced which include the possibility of applying a thickness up to 4-6mm. They have emerged with the aim of reducing the polymerisation shrinkage while simplifying the insertion process by placing layers of larger thickness. This takes less time than the traditional approach and is especially useful in deep cavities of posterior teeth. This is made possible by the many specificities of these composite resins, which makes them perfect for treating posterior teeth. With photo initiators enabling a deeper polymerisation and polymerisation modulators permitting less polymerisation shrinkage, they exhibit higher translucency and, as a result, better light dissipation in the composite resin.

The higher mobility of monomers caused by the increased temperature facilitates crosslinking between polymer chains and results in a high conversion degree [2]. Preheating composite resin decreases its viscosity and thickness, increasing its flow and adaptation with the cavity walls [3]. Monomers can attain lower viscosity when they are warmed. This is based on the theory that thermal energy forces the monomers or oligomers to move apart, allowing them to slide by each other more easily. The heat induced by the preheated composite resin has a minimal effect on the temperature rise of the pulp (approximately 2°C) and can be tolerated by the pulp [4] without causing any damage.

Pulp can withstand high temperatures. Nevertheless, it is not clinically advisable to heat the composite resin above 60 °C as it can cause irreparable damage to the pulp [5] Demirel et al. investigated the influence of different insertion techniques on internal pore and void formation, and later found that the application of a thermoviscous

bulk-fill composite with the pre-heating technique showed the least void percentage [6].

Hence objectives of this study include:

□ To evaluate the microhardness of preheated bulkfill and conventional composite resins

□ To find out which bulkfill composite has better performance while preheating.

□ To find out whether preheating enhances the performance of conventional composites

MATERIALS AND METHODS

1) Ivoclar Tetric N-Ceram bulkfill

2) 3M Filtek bulkfill

3) Tokuyama Estelite Alpha

4) Dentsply Sirona Neospectra ST Universal composite

5) Teflon moulds

6) Composite preheating device

7) Digital thermometer

8) Hand tools

9) LED Curing lamp

10) Glass slab and glass slide

11) Grit silicon carbide polishing sand papers

12) Vickers hardness indenter

STUDY METHOD

Three commercial bulkfill resin based composite materials such as Ivoclar Tetric N-Ceram Bulkfill, 3M Filtek Bulkfill, Tokuyama Estelite Alpha bulkfill and one conventional composite resin- Dentsply Sirona Neospectra ST Universal composite resin of A3 shade was evaluated in this study. All the chosen composites were of high viscosity-packable in consistency. n=6 samples were produced using Teflon moulds measuring 2mm & 4mm thickness and 8mm in diameter. Half of the samples from each composite category, underwent preheating, while the remaining half were made at room temperature. The ventilation system of the laboratory managed the temperature variation. A digital thermometer was used to check the room temperature.

Composite syringes or capsules were warmed in batches using a composite heating apparatus named Endoking Nanowarm Composite Warmer at a consistent temperature of 50°C for 10 minutes. All warmed composite materials were inserted into the acrylic mould within 45 seconds of being taken out from the heating apparatus. Subsequently, the samples were cured using an LED light device that emitted a high-intensity 430-480nm wavelength and 1200 mW/cm² light power for 20 seconds. For more accuracy, intensity of the light was monitored using a radiometer and was made constant within the mentioned range throughout the procedure. To standardize the distance of the light reaching the samples, the LED device was positioned close to the glass slide on the top surface, and polymerization was accomplished.

POLISHING OF COMPOSITE SAMPLES:

All samples were polished with 800, 1000 grit silicon carbide sand paper, respectively, in circular motions with finger pressure for a duration of 3 minutes. Silicon carbide paper was utilised to standardize and remove the oxygen inhibition layer. The polishing process was done by a single operator. All the

samples were made smooth with unused fresh sand papers to obtain maximum efficiency and to make sure that all the samples have the same level of polishability.

HARDNESS TESTING (DATA COLLECTION):

Vickers hardness test was utilized to analyse the microhardness of the samples. The samples were placed on the table of the test device, and a 100-gram (980.7mN) load was put on to the sample for 15 seconds. The Vickers Hardness Number (VHN) was obtained from the top and bottom surfaces of the samples at three equally spaced points using millimetric paper. Following each measurement, the samples were kept in distilled water in a separately numbered box. This is done in order to mimic the moist environment of the oral cavity. VHN values were determined instantly after preheating the samples, and after 24-hour storage period in distilled water at room temperature. The values on the top and bottom surfaces were estimated by taking the average of the three measured points

Ethical Approval: The ethical clearance was waived by the Institutional research and ethical committee of Kannur Dental College, Kerala.

Study setting:

1) Kannur dental college, Kerala, India

2) Mechanical testing lab at National Institute of Technology, Calicut, Kerala, India

Statistics

Sample size calculation was done using mean microhardness of bulk fill and conventional composites from a previous similar study.

$$n = (Z\alpha/2 + Z1-\beta)^2 \times 2 (SD)^2 / d^2$$

d =

SD= Pooled SD

d= difference in mean

Z $\alpha/2$ = Normal deviate for two tailed hypotheses = 1.96

Z1- β = 0.84

According to a previous study, Degirmenci and Can, microhardness of bulk-fill (3M Filtek) and conventional composites were 38.1 $\pm$ 1.7 and 31.3 $\pm$ 2.1 respectively. Pooled standard deviation and mean difference from the study was used to calculate sample size for the current study.

Formula for Pooled standard deviation, $SD = \sqrt{((SD_1^2 + SD_2^2) / 2)}$

$$SD = \sqrt{((1.7^2 + 2.1^2) / 2)} = 2$$

Difference in mean, d=6.8

$$n = ((1.96 + 0.84)^2 \times 2 \times 2) / 6.8^2$$

Substituting these values in the formula, calculated minimum sample size in a group for conducting the study before dropout was 2. Rounding of and adjusting sample size of 6 can be taken in each group.

STUDY GROUPS

GROUP I: Preheated and non-preheated

GROUP II: 2mm and 4mm thickness application

GROUP III: Baseline and 24 hours

STATISTICAL ANALYSIS

The data was analysed using IBM SPSS V.25 software. The data was cleaned and coded in SPSS and the normality and presence of outliers were assessed using Shapiro Wilk test of normality and box plots. The Descriptive tests were followed by generalised linear model method to examine the

effects of material, heating and time on the microhardness values. Repeated measure ANOVA comparing mean scores across multiple observations was planned if the values following normal distribution. It evaluates differences over time or conditions, unlike standard ANOVA. If the data is not following a normal distribution, Friedman Test will be done. The p values less than 0.05 was considered significant.

RESULTS

Descriptive statistics were used to summarize the microhardness measurements. The distribution of samples across the experimental groups is presented in Table 2. Mean VHN values for the 2-mm and 4-mm specimens, according to material, preheating condition, surface, and time of testing, are presented in Tables 3 and 4, respectively. Repeated-measures analysis was used to evaluate changes across experimental conditions.

The VHN values differed across the tested materials and experimental conditions. Preheating was associated with an increase in VHN in several comparisons involving 3M Filtek Bulk Fill, Ivoclar Tetric N-Ceram Bulk Fill, and Dentsply Neospectra, whereas Tokuyama Estelite Alpha showed a different response in some comparisons. The magnitude and direction of change also depended on specimen thickness, surface, and time of testing.

At baseline, the numerical values presented in Tables 3 and 4 show that 3M Filtek Bulk Fill had comparatively higher VHN values than Tokuyama Estelite Alpha. Following 24 hours of storage in distilled water, 3M Filtek Bulk Fill continued to show comparatively high VHN values in the reported conditions, while the change from baseline to 24 hours varied among materials. Therefore, the previously stated reversal in which Tokuyama was described as having the highest baseline VHN and 3M Filtek the lowest has been removed because it was inconsistent with the tabulated VHN values.

The statistical model indicated that VHN was influenced by the experimental factors and their interaction. Exact inferential statistics should be reported from the verified original statistical output when available; therefore, no new exact p-values have been generated from summary values alone. Comparisons of microhardness according to material, surface, preheating process, and time are presented in the corresponding tables and graphs.

DISCUSSION

Today, improvements in formulations, optimization of properties and the development of new techniques for placement have made the restoration of direct composite more reliable and predictable. Producers tried to decrease the viscosity of dental composite to make either the material or the technique easier to apply and faster to use. Several solutions have been proposed to decrease composite viscosity [7].

Pre-heating and the consequential rapid cooling may differently alter the polymerization kinetics of resin composites depending on their composition and manipulation method. Thus, the final network configuration and the arrangement of the connected and unconnected monomer units could be different. In addition to the setting kinetics, the application technique may also influence pore formation, leading to a change in the amount of leachable unreacted monomers [6]. Although preheating conventional or bulk-fill RBCs(

resin-based composites) has been extensively investigated, there is no available literature about how pre-heating affects the depth of cure of chosen composites, which are important factors with regard to mechanical properties.

In this study, the microhardness of preheated and non-preheated bulk-fill composite materials was compared with that of a conventional composite resin at baseline and after 24 hours of storage. The effect of preheating was material-dependent and also varied according to specimen thickness and surface. Based on the VHN values reported in Tables 3 and 4, 3M Filtek Bulk Fill demonstrated comparatively higher hardness values, whereas Tokuyama Estelite Alpha generally showed lower values. Changes between baseline and 24 hours were not uniform across all materials. These findings indicate that the influence of preheating on microhardness should be interpreted according to the specific resin composite and experimental condition rather than as a uniform effect across all materials.

According to the results of this in vitro study, preheating process increased the microhardness values of some of the bulkfill composite samples particularly composite 3M group which contained highly viscous monomer such as Bis-GMA. The composition of the tested composite resins affected their microhardness values.

On the contrary to the results of this study, Bhopatkar et al in his study of assessing surface microhardness of various composite materials after preheating found Ivoclar Tetric N-Ceram Bulk Fill to have the greatest value at a preheating temperature of 60°C [9].

Conversely, other studies [10–12] reported that preheating did not affect the microhardness of resin composites. This finding might be attributed to the residual stresses that can be generated due to elevated temperatures. Residual stress is a form of concentrated energy in the bulk of the material without the application of an external load. When the composite resin restoration experiences an occlusal load, the microhardness decreases and the bonding failure increases.

Kusgoz et al. claimed that this peculiar initiating system could be responsible for the lower depth of cure of silorane-based composites in comparison to methacrylate-based composites. Consequently, the reduced depth of cure of silorane based composites is reflected in their reduced hardness [13].

Increased temperature decreases viscosity, increases free radical mobility, and increases the frequency of conflicts between the active group and free radicals. As a result, the degree of conversion is improved. This is expressed by the increased microhardness of the composite resin.

Under clinical conditions, two important factors involved in preheating of composite resins are:

- The temperature of composite when placed in the cavity
- The time interval between removal of the composite resin from the compoule and its placement in the cavity.

Theoretically, thermal vibration causes the resin monomers or oligomers to split, making them simpler to slide between one another, which results in composite resins having reduced viscosity. The fluidity of the composite resin is improved by preheating it. It also becomes more adaptable to the cavity wall and has better edge closure, which reduces microleakage. After a

filling, microleakage of the edges can make teeth more sensitive, change the color of the edges, and possibly lead to secondary caries and loose shedding of restorations.

The heat that is generated during polymerization and the thermal expansion-coefficient mismatch between the polymer matrix and the fillers can result in stress development at the filler-matrix interface, which can generate internal 'hoop' stresses around the fillers¹⁵.

Tantbirojn et al. discovered that the stress caused by the composite resin's polymerization contraction can deform the tooth tissue or restoration, leading to adhesion failure and significantly reducing the resin filling repair long-term effectiveness⁵.

The degree of conversion is a criterion for determining the mechanical qualities of restorative materials at the end of the process. Preheating composite resins has been proposed as a method of increasing the degree of conversion and, as a result, microhardness, wear tolerance, and fracture resistance. However, based on the results of their study, if composite resin is cooled to 40°C, its advantages can still be helpful compared to composite resin cooled to room temperature.⁵

In order to achieve best clinical performance, the clinician must work very quickly to ensure the least temperature drop possible. The dispensed material should be placed, adapted, contoured, and light-cured in minimal time to attain potential added conversion above that of room temperature values. Additionally, a longer heat soak might be necessary to reduce the cooling effect when a composite compoule is removed from the warmer unit.¹⁶ Moreover, the rapid cooling of the pre-heated RBC before polymerization may contribute to the structural heterogeneity of the polymer network⁶.

The clinical concern is that if preheated composite is not used immediately, degradation of resin components or premature curing of the materials would occur. Under the extreme conditions of temperature storage and cycling imposed during this testing, it can be concluded that exposure of sealed compoules to such thermal insults does not degrade their ability to polymerize.

Increased polymerization temperature increases conversion of dimethacrylate monomers, but only up to a certain temperature limit. After that limit, monomer conversion decreases with subsequent temperature increase. For monomers such as Bis-GMA or Bis-EMA, this limit occurs near 90°C. Because elevated temperatures may volatilize reactive components, it should be stressed that compoule caps and plungers should be checked for security before and subsequent to heating (if reused).¹⁶

MANUFACTURER INFORMATION OF THE COMPOSITE MATERIALS USED

Table 1

MAT ERI AL	MANUF ACTUR ER	CLASS IFICA TION	COMPOSITION
NEO SPEC TRA ST	Dentsply Sirona, U SA	Nanohy brid	Bis- GMA, TEGDMA, UDMA, submicron filler technology <1µm, spherical, pre-polymerized SphereTEC® fillers (d3,50≈15

In a study by Mehdi Abed et al, color changes were evaluated after 40 cycles of preheating, and significant differences were detected. So, repeated cycles of preheating harm the color stability of composite resins¹⁷. The current study has some limitations. In this study, the researchers considered standardizing the specimens by applying different composite resin materials in a single layer to obtain uniform thickness. At the same time, the specimens were polymerized only from the top side to mimic the clinical situation. This means that polymerization has less effect on the behaviour of the material, and therefore, the filler particle type and content (percentage) play a greater role. Additionally, long-term clinical studies are recommended to evaluate the clinical performance of these materials.

CONCLUSION

Based on the results of laboratory studies, preheating procedures for dental restorative materials is a simple, safe, and relatively successful technique. In general, for resinous materials, there is an increase in microhardness and degree of conversion, reduction in viscosity, and better adaptation to cavity walls. In order to achieve good results, agility and training are necessary so the material would not lose heat until the restorative procedure. Also, care is necessary to avoid bubbles and formation of gaps, which compromises the best restoration performance⁸.

CONFLICT OF INTEREST

Nil

AUTHOR'S CONTRIBUTIONS

Conceptualization, first author
methodology, second author
software, third author
validation, fourth author
formal analysis, fifth author
investigation, sixth author
resources, first author
data curation, second author
writing—original draft preparation, third author;
writing—review and editing, fourth author
visualization, fifth author
supervision, sixth author
project administration, first author;
funding acquisition, second author

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3M, USA

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			µm), non-agglomerated barium glass and ytterbium fluoride.
ESTE LLIT E ALP HA	Tokuyama dental corporations, Tokyo, Japan	Nanohybrid	Bis-GMA, TEGDMA, 82 % by weight (71 % by volume) of submicron (0.2µm) Silica-zirconia spherical filler and composite filler

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TETRIC N-CERAM BULK FIL	Ivoclar Vivadent inc.	Nanohy brid	Dimethacrylates(B is-GMA,UEMA,UDMA),Barium glass,Yttrium trifluoride,mixed oxide and prepolymer,additives,catalysts,stabilizers,pigments
FILTEK BULK FIL	3M ESPE, St. Paul, MN, USA	Nanohy brid	AUDMA, UDMA, 1,12-dodecane-DMA non-agglomerated/non-aggregated 20-nm silica filler, non-agglomerated/non-aggregated 4- to 11-nm zirconia filler, aggregated zirconia/silica cluster filler (comprised of 20-nm silica and 4- to 11-nm zirconia particles), ytterbium trifluoride filler consisting of agglomerate 100-nm particles

TABLE 2

	Sample group	No. of samples in the group (N=96)
	2 mm application	
1	Preheated Bulkfill 3M	6
2	Preheated Bulkfill TY	6
3	Preheated Bulkfill Ivoclar	6
4	Preheated Conventional	6
5	Non- Preheated Bulkfill 3M	6
6	Non-Preheated Bulkfill TY	6
7	Non-preheated Bulkfill Ivoclar	6
8	Non-preheated Conventional	6
	4 mm application	
9	Preheated Bulkfill 3M	6
10	Preheated Bulkfill TY	6
11	Preheated Bulkfill Ivoclar	6
12	Preheated Conventional	6
13	Non-Preheated Bulkfill 3M	6

14	Non-Preheated Bulkfill TY	6
15	Non-preheated Bulkfill Ivoclar	6
16	Non-preheated Conventional	6

TABLE 3

	2MM											
	EXPOSURE TO HEAT											
	NON-PREHEATED						PREHEATED					
MATERIAL	TOP HARDNESS	BOTTOM HARDNESS	MEAN	TOP HARDNESS	BOTTOM HARDNESS	MEAN	TOP HARDNESS	BOTTOM HARDNESS	MEAN	TOP HARDNESS	BOTTOM HARDNESS	MEAN
	BASELINE	24 HRS	BASELINE	24 HRS	BASELINE	24 HRS	BASELINE	24 HRS	BASELINE	24 HRS	BASELINE	24 HRS
BULK FILL - 3M	103.79	92.36	91.33	77.58	97.47	84.72	106.74	96.12	103.43	97.73	105.27	95.77
BULK FILL - TOKUYAMA	45.77	39.21	36.54	30.19	43.57	34.99	40.77	33.57	32.07	36.77	33.77	31.77

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B U L K F I L L - I V O C L A R	5 6 . 3 4	4 2 . 7 6	4 8 . 5 4	3 3 . 7 8	5 2 . 4 4	4 7 . 6 6	5 1 . 6 6	4 8 . 0 5	4 7 . 0 1	4 2 . 7 6	4 9 . 3 3	4 5 . 4
C O N V E N T I O N A L - D E N T S P L Y	5 2 . 2 7	3 2 . 2 3	3 6 . 0 6	2 4 . 1 7	4 4 . 1 6	2 8 . 2	5 6 . 3 4	5 8 . 5 7	4 7 . 3 2	5 0 . 3 7	5 1 . 8 3	5 4 . 4 7

Source	Degree of freedom	Sig.
Material	3	<0.001***
Time	1	<0.001***
Heat	1	0.708

TABLE 4

	4MM	
	EXPOSURE TO HEAT	
	NON- PREHEATED	PREHEATED

M A T E R I A L	T O P H A R D N E S S		B O T T O M H A R D N E S S		M E A N		T O P H A R D N E S S		B O T T O M H A R D N E S S		M E A N	
	B A S E L I N E	2 4 H R S	B A S E L I N E	2 4 H R S	B A S E L I N E	2 4 H R S	B A S E L I N E	2 4 H R S	B A S E L I N E	2 4 H R S	B A S E L I N E	2 4 H R S
B U L K F I L L - 3 M	104. 98	94. 04	95. 53	81. 81	100. 25	87. 92	109. 34	101. 76	103. 87	98. 70	106. 6	100. 23
B U L K F I L L - T O K U Y A M A	56. 86	68. 44	46. 05	54. 06	51. 45	61. 25	43. 05	44. 93	37. 02	37. 74	40. 03	41. 33
B U L K F I L L - I V O C L A R	58. 83	50. 22	48. 82	42. 68	50. 75	46. 45	53. 41	51. 68	48. 60	42. 37	51	47. 02

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C	39.	28.	25.	19.	32.	24.	48.	40.	37.	32.	42.	36.
O	45	43	24	61	34	02	02	34	45	27	73	3
N												
V												
E												
N												
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Figures

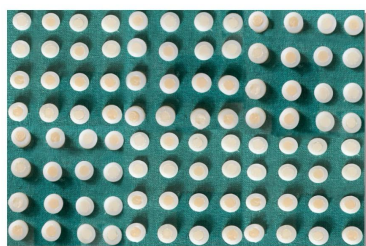


Figure 1 : n=6 samples were produced using Teflon moulds measuring 2mm & 4mm thickness and 8mm in diameter

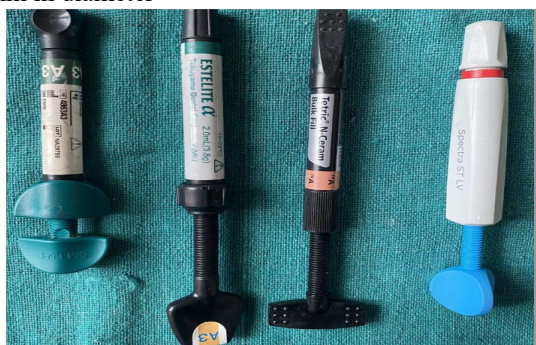


Figure 2: Three commercial bulkfill resin based composite materials such as Ivoclar Tetric N-Ceram Bulkfill, 3M Filtek Bulkfill, Tokuyama Estelite Alpha bulkfill and one conventional composite resin- Dentsply Sirona Neospectra ST Universal composite resin of A3 shade was evaluated in this study. All the chosen composites were of high viscosity-packable in consistency.



Figure 3: A digital thermometer was used to check the room temperature



Figure 4: Composite syringes or capsules were warmed in batches using a composite heating apparatus named Endoking Nanowarm Composite Warmer at a consistent temperature of 50°C for 10 minutes

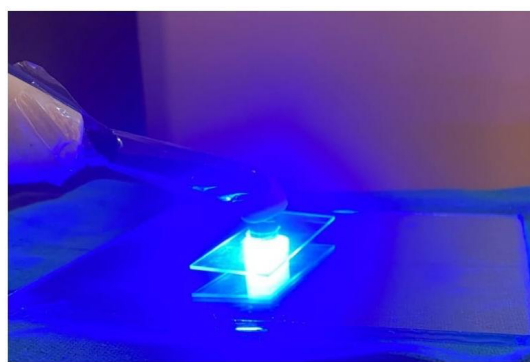


Figure 5: Samples were cured using an LED light device that emitted a high- intensity 430-480nm wavelength and 1200 mW/cm² light power for 20 seconds

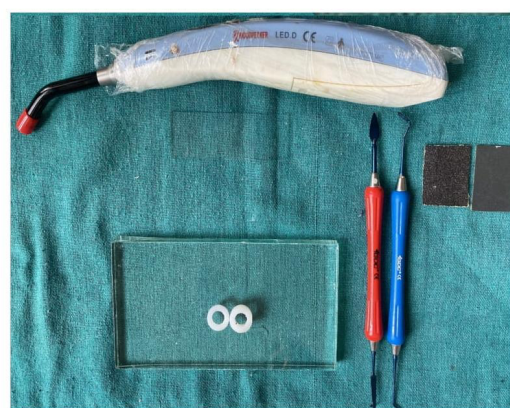


Figure 6: To standardize the distance of the light reaching the samples, the LED device was positioned close to the glass slide on the top surface, and polymerization was accomplished



Figure 7: The samples were placed on the table of the test device, and a 100-gram (980.7mN) load was put on to the sample for 15 seconds

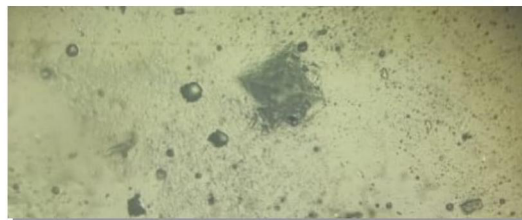


Figure 8: The values on the top and bottom surfaces were estimated by taking the average of the three measured points

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