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Comparative Evaluation of the Degree of Polymerization of Three Different Light Sources Used for Orthodontic Bonding: An in-Vitro Study

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I. Introduction

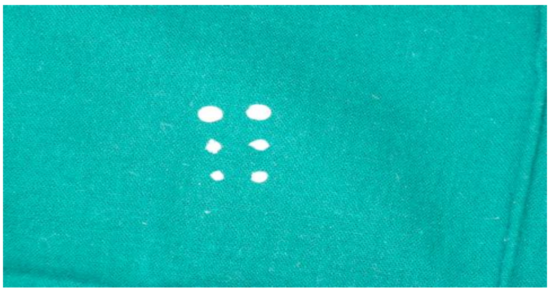
Evolution has been in progress from the time light cure adhesives came into clinical use in orthodontics. The ongoing search for methods to enhance polymerization and reduce curing time was always given priority, which triggered the emergence of new light curing devices. In recent years, research and development in this domain has resulted in identifying several new concepts in curing systems owing to the disadvantages faced by using halogen units such as, reduced life of the bulbs, increased curing time and large unit size. In 1995, Light Emitting Diode (LED) was proposed as an excellent polymerization source. [1,2] The LED curing unit proved more efficient than conventional halogen lights as they possessed high curing efficiency, higher intensity, shorter curing time and comparable bond strength. These lights have an output of 1000 mW/cm^2 and were capable of curing the brackets in 10-20 seconds. The recently marketed newer generation high intensity LED curing light has a prescribed curing time of 3 sec mesial and distal with no compromise on bond strength, as claimed by the manufacturer.[3] The peak wavelength being 455 nm and output 1600 mW/cm^2 , thus the reduction in curing time. [4] A hypothesis that stated the different types of light-curing sources when used for the recommended irradiation times, resulted in different % Degree of Conversion.[5]

The Degree of polymerization or conversion of a resin composite is defined as the percentage of reacted C=C bonds. This ratio substantially affected many properties including mechanical properties, solubility, dimensional stability, color change and biocompatibility of the resin composite. Thus, the degree of conversion played an important role in determining the ultimate success of a light activated resin. Work by Emami et al in 2003 [6] had shown a close correlation between energy density and degree of conversion. The increased energy densities lead to superior physical and mechanical properties. Markus Niepraschk et al (2007) [7] showed that the LED and the halogen lights, with the 20-second regimen, produced higher %DC (Degree of Conversion). No significant difference was found in %DC between the plasma light and the 10-second LED light, whereas both showed lower %DC compared to 20 seconds of halogen and LED light. Earlier studies have thrown more light on the changes in degree of conversion by altering the wavelength, energy density and the prescribed curing time of the various light sources. Little evidence was available regarding the curing efficiency of the light sources when the composite thickness was varied.

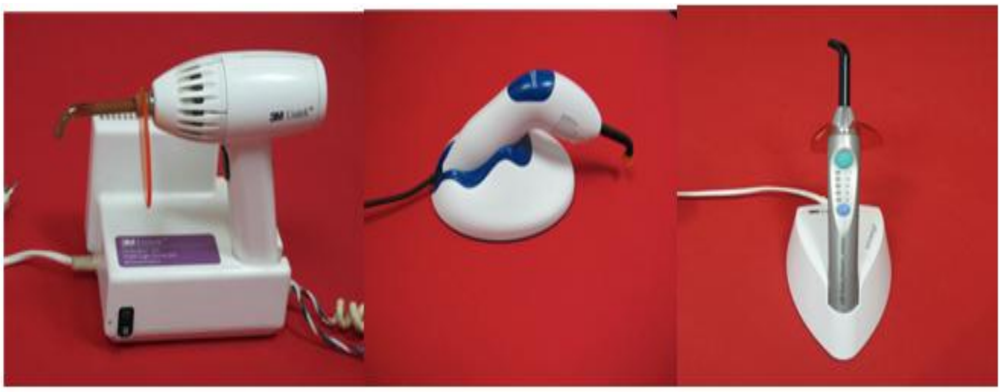
The varying thicknesses of composite used in lingual orthodontics up to 2mm to offset the brackets, gave us reason to speculate whether the light source used had efficiently cured the composite to an acceptable uniformity. Hence, this study was proposed to evaluate the degree of polymerization of composite of three varying thicknesses with the three different light sources. The null hypothesis states that the degree of polymerization is unaltered with varying thickness of composite cured using different intensities of light source.

II. Materials And Methods

The samples used were pellets of composite resin in varying thicknesses of 0.5mm, 1mm and 1.5mm cured using the three different light sources.



The types of light sources that have been used in this study are Halogen, Conventional LED and Luminous fast-curing LED unit.



The curing times were 40 sec, 20sec and 3 sec respectively, according to the manufacturers' prescription. The distance maintained between the tip of the light curing units and the adhesive resin was 1mm. [8, 9]

Grouping of samples

Fifteen samples of 0.5mm, 1mm and 1.5mm thicknesses of light cure resin were cured with halogen light for 40 seconds.

The samples were then placed in separate labeled covers as Group Yellow (A) under subgroups of 0.5mm, 1mm and 1.5 mm thicknesses.

The same procedure was repeated for the conventional LED and the samples were placed in labeled covers as Group Red (B) with subgroups of 0.5mm, 1mm and 1.5mm thicknesses.

Similarly samples were made for Group Green (C) cured with the luminous fast-curing LED device with subgroups of 0.5mm, 1mm and 1.5 mm thicknesses.

Sample preparation for cured resin

The sample pellets were made using three transparent rubber moulds with inner surface depth of 0.5mm, 1mm and 1.5mm.

Group (A) samples were prepared by filling Transbond XT light cure resin in the rubber mould of 0.5mm depth. Cover slip was placed on it and cured with the halogen light for 40 seconds from a distance of 1mm from the composite. Once cured the 0.5 mm pellet was placed in the labeled cover.The same procedure was repeated for the 1mm and 1.5mm samples as well. The Group (B) and Group (C) were prepared similarly and cured with the conventional LED and the new luminous fast-curing LED respectively and placed in their labeled covers.Fifteen samples were made under each subgroup in the same designated manner.They were stored in a dry place at 370 ^{0}C for 24 hours.

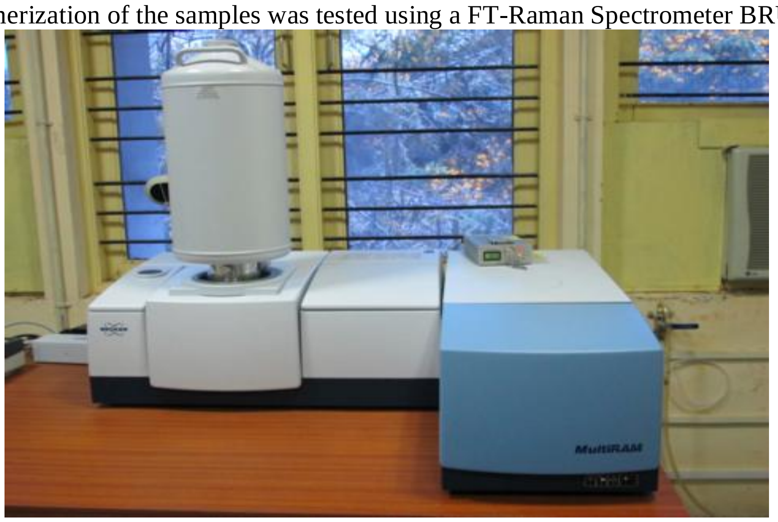
Sample preparation for uncured resin

The uncured sample of the light cure resin was filled in the depression present in the circular metal holder specific to the FT-Raman instrument.

The actual depth of the depression which was present at the centre of the metal holder is 1mm. The light cure resin was filled to 0.5mm and 1mm for the first and second sample. For preparing the sample of 1.5mm, the resin was allowed to flow 0.5mm out of the depression and measured accurately with a scale before testing. The resin was filled in different metal holders for the purpose of the study.

Procedure for Degree of polymerization

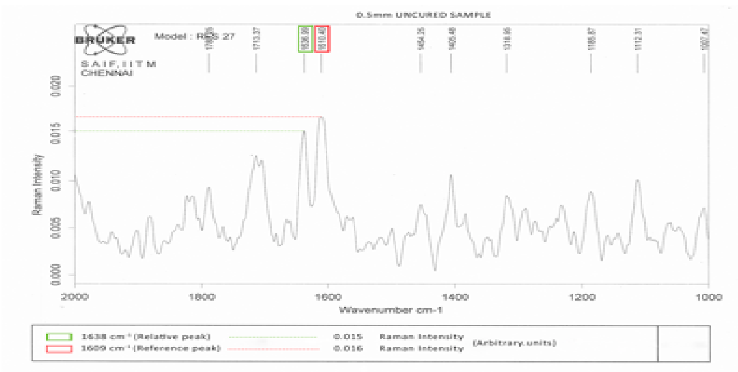
The degree of polymerization of the samples was tested using a FT-Raman Spectrometer BRUKER RFS 27.



The laser source was Nd: YAG 1064 nm. In order to study the degree of polymerization of the uncured and cured resin samples with their respective thicknesses, they have to be subjected to the FT-Raman spectrometer to acquire the relative and reference peak intensities.

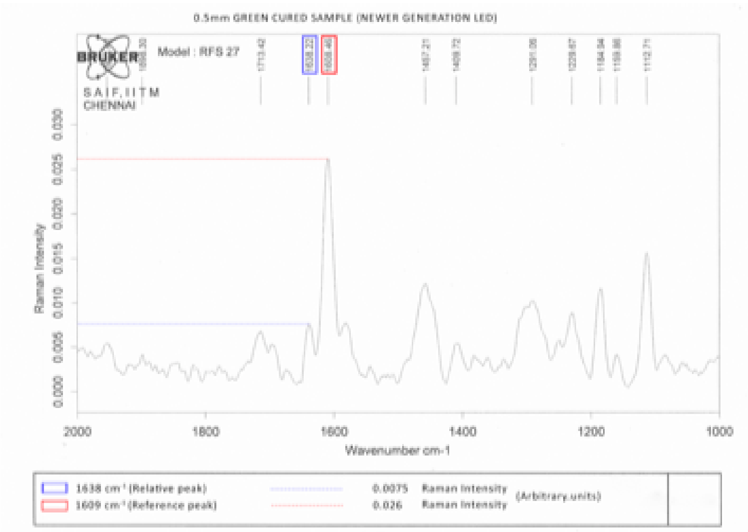
Degree of polymerization measurements

The samples of uncured resin thus prepared were kept in the FT-Raman instrument and were tested to find out the reference and relative peak intensities. A graph was recorded for each sample.



The cured sample of Group (A) subgroup 0.5mm was placed on the metal holder of the Raman spectroscopy, with the surface of the pellet facing the curing light.

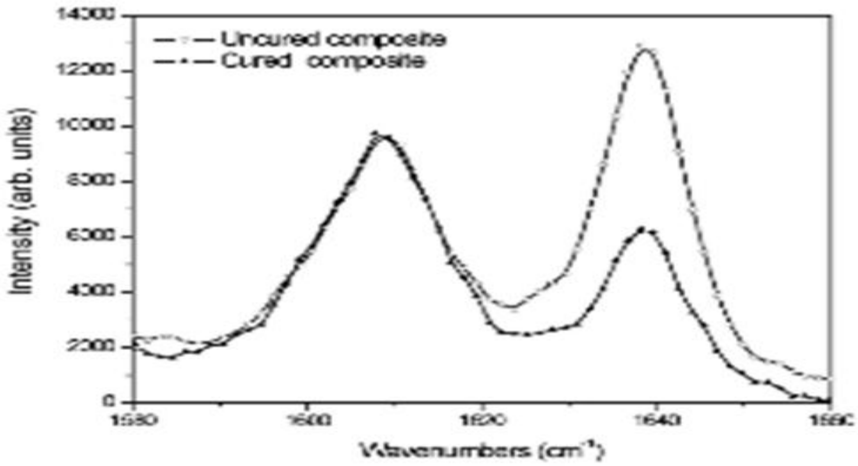
Then the laser light of wavelength 1064 nm was passed through the sample which excited the sample. This was repeated for the 15 samples of 0.5mm, 1mm and 1.5 mm thickness and the samples were tested for the degree of polymerization. The light from the laser was scattered in all directions and directed towards the detector which records the Raman Spectrum.[10] This spectrum showed Raman spectral features unique to the sample. A graph was generated with peaks and the corresponding intensities of the peaks (1609 cm^{-1} being the reference peak and 1638 cm^{-1} the relative peak)[11] for Bis-GMA present in the resin, were taken for each sample and substituted in the formula.



(Transbond XT that we used in this study is composed of 14% volume Bis-GMA, 9% volume Bis-EMA and 77% volume filler particles).

Calculation of Degree of conversion

The calculation of the degree of polymerization was based on the measurement of the net absorbance peak of the aliphatic bond stretching vibrations of the C=C bond (1638 cm^{-1}) taken as the relative peak affected by polymerization and the aromatic C-C bonds (1609cm^{-1}) taken as the reference peak as it was not affected by polymerization.[12] The net absorbance peak area ratio of cured to uncured material provides the percentage of converted double bonds.



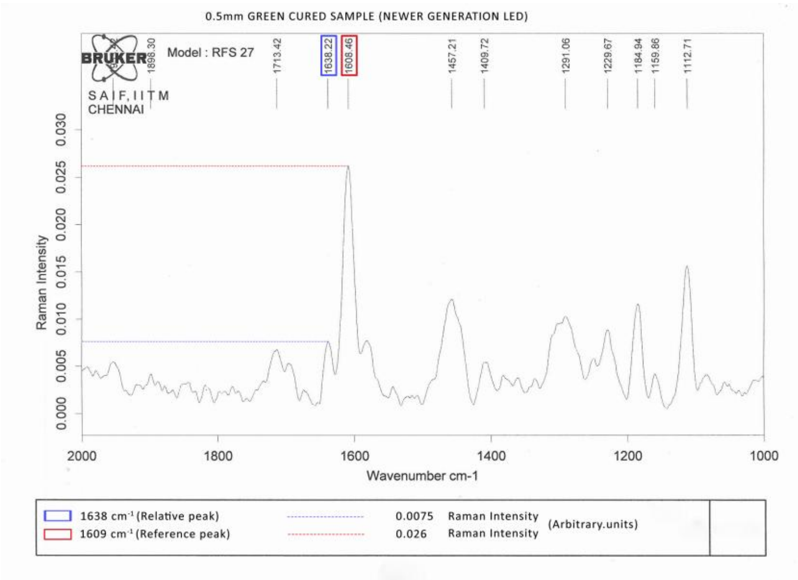
The peak recorded at 1609 cm^{-1} was the reference peak and its intensity was not affected by curing. The peak recorded at 1638 cm^{-1} was the relative peak and its intensity reduced when the resin was cured. The arbitrary intensity values were substituted in the formula to calculate the degree of conversion.

The degree of conversion percentage (DC%) was calculated from the ratio between the peaks of the aliphatic C=C bond (1638 cm^{-1}) to the aromatic C-C bond (1609cm^{-1}) obtained from the cured and uncured specimens using the following equation [12] :

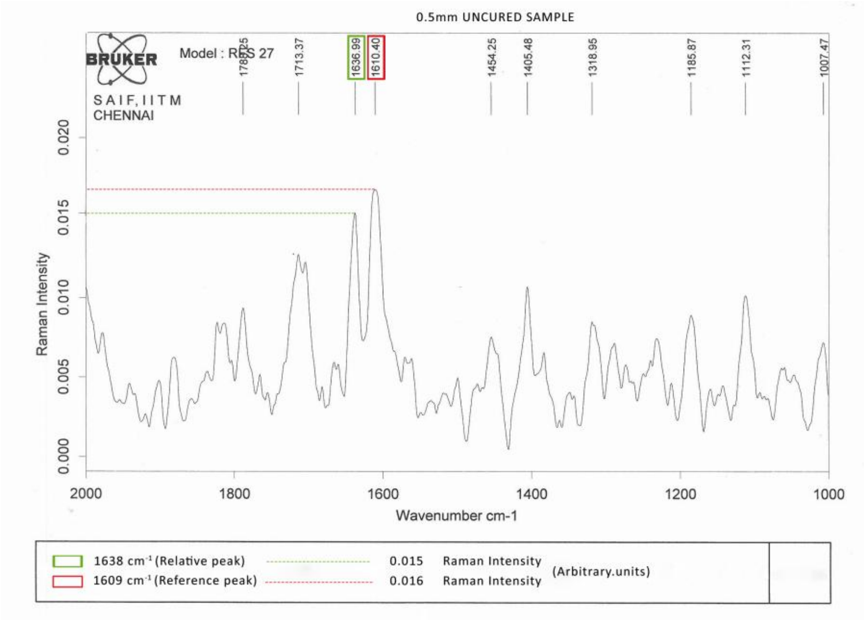
$$DC(\%) = \left\{ 1 - \left[\frac{R_{polymerized}}{R_{unpolymerized}} \right] \right\} \times 100$$

R = Relative peak(1638 cm^{-1}) / Reference peak(1609 cm^{-1})

The graph for 0.5mm cured resin



The graph for 0.5mm uncured resin



Residual double connections (%) =

$$\frac{\text{Absorbance in } 1638\ cm^{-1}/\text{absorbance in } 1608\ cm^{-1}\text{(Polymerized)}}{\text{Absorbance in } 1638\ cm^{-1}/\text{absorbance in } 1608\ cm^{-1}\text{ (Non-Polymerized)}} \times 100$$ [13]

Degree of conversion (%) = 1 – Residual double connections (%)

The Raman intensity peak values were arbitrarily taken from the graph and substituted in the formula:

$$\frac{0.0075/0.026}{0.015/0.016} = \frac{0.288}{0.937} = 0.307$$

Degree of conversion (%) = 1 – Residual double connections (%)
Degree of conversion (%) = 1 - 0.307 = 0.69

Degree of Polymerization in percentage = 0.69 x 100= 69%

So the degree of conversion = 69% for the 0.5 mm sample cured with the newer generation LED.

The calculation was applied for all the subgroups within each group and compared for difference in percentage degree of polymerization.

	Halogen light			Conventional LED			Newer generation LED		
sample	0.5mm	1mm	1.5mm	0.5mm	1mm	1.5mm	0.5mm	1mm	1.5mm
1	69.00%	61.00%	57.00%	64.00%	63.00%	48.00%	69.00%	55.00%	49.00%
2	68.00%	60.00%	55.00%	62.00%	61.00%	47.00%	68.00%	54.00%	45.00%
3	66.00%	59.00%	57.00%	64.00%	63.00%	47.00%	69.00%	58.00%	49.00%
4	69.00%	61.00%	57.00%	64.00%	63.00%	45.00%	69.00%	55.00%	49.00%
5	65.00%	61.00%	56.00%	65.00%	62.00%	48.00%	69.00%	55.00%	49.00%
6	69.00%	61.00%	57.00%	64.00%	63.00%	46.00%	67.00%	57.00%	49.00%
7	69.00%	67.00%	57.00%	64.00%	63.00%	48.00%	69.00%	55.00%	49.00%
8	67.00%	61.00%	56.00%	63.00%	63.00%	47.00%	69.00%	55.00%	49.00%
9	69.00%	61.00%	57.00%	64.00%	59.00%	48.00%	69.00%	55.00%	49.00%
10	69.00%	64.00%	57.00%	65.00%	63.00%	48.00%	69.00%	56.00%	49.00%
11	69.00%	61.00%	57.00%	64.00%	63.00%	47.00%	66.00%	55.00%	49.00%
12	68.00%	61.00%	57.00%	64.00%	62.00%	48.00%	69.00%	55.00%	49.00%
13	69.00%	61.00%	55.00%	66.00%	63.00%	46.00%	69.00%	58.00%	49.00%
14	69.00%	63.00%	57.00%	64.00%	63.00%	48.00%	69.00%	55.00%	49.00%
15	69.00%	61.00%	53.00%	64.00%	62.00%	47.00%	69.00%	57.00%	49.00%

III. Statistical Analysis

ANOVA and Post Hoc Tests were used to compare the percentage degree of polymerization of the groups and within the groups. Significance for all statistical analysis were performed with SPSS VERSION 15

IV. Results

The results of the analysis of variance (ANOVA) for groups 0.5mm, 1mm and 1.5mm are shown in (Table 2, 3 and 4) . This revealed the presence of significant differences among the groups. The Statistical significance is high ($p<0.05$) between Groups A and B, Groups A, C and Groups B, C.

O.5mm

Groups	No. of samples	Mean (%)	Std deviation	Significance
A -Halogen	15	68.27	1.28	0.000
B -Conventional LED	15	64.07	0.88	
C -New luminous curing light	15	68.60	0.91	

1mm

Groups	No. of samples	Mean (%)	Std deviation	Significance
A -Halogen	15	61.53	1.88	0.000
B -Conventional LED	15	62.40	1.12	
C -New luminous curing light	15	55.66	1.23	

Groups	No. of samples	Mean (%)	Std deviation	Significance
A -Halogen	15	56.33	1.17	0.000
B -Conventional LED	15	47.20	0.94	
C -New luminous curing light	15	48.73	1.03	

V. Discussion

This study was performed to evaluate and compare the efficacy of light sources used to polymerize different thicknesses of composite (0.5mm, 1mm and 1.5mm). In clinical situations involving lingual orthodontics, composite thickness was varied on the bracket bases to offset them to a maximum of 2mm for efficient aligning and complete wire insertion. This study was done to evaluate whether the degree of polymerization (55-75%) [11] was adequately achieved in such special clinical situations, when using newer generation light sources with reduced curing time. The study was conducted on 135 pellets of light cure composite resin (Transbond XT, 3M Unitek) of varying thicknesses of 0.5mm, 1mm and 1.5mm cured using Halogen, Conventional LED and Newer generation LED. The distance between the light source and resin composite directly affected light intensity on the resin surface. Light intensity diminished when the distance of the tip to the resin composite was increased. Thus, the most common clinical recommendation for the position of the light curing appliance tip was maintained at 1 mm from the resin.[14, 15]

Group A comprised of 45 samples divided into subgroups of 0.5mm, 1mm and 1.5mm consisting of 15 samples each, cured using halogen light. Group B comprised of 45 samples divided into subgroups of 0.5mm, 1mm and 1.5mm consisting of 15 samples each, cured using conventional LED. Group C comprised of 45 samples divided into subgroups of 0.5mm, 1mm and 1.5mm consisting of 15 samples each cured using the Newer generation luminous curing light.

The mean degree of polymerization was evaluated for Groups A, B and C for the thickness of 0.5 mm. The subgroup 0.5mm of Group A showed percentage degree of polymerization of 68.27 ± 1.2% with a maximum value of 68.97 and minimum of 67.56. The subgroup 0.5mm of Group B showed percentage degree of polymerization of 64.07 ± 0.88% with a maximum value of 64.56 and minimum of 63.58. The subgroup 0.5mm of Group C showed percentage degree of polymerization of 68.60 ± 0.91% with a maximum value of 69.10 and minimum of 68.09.Comparing the mean percentage degree of polymerization, Group C had the highest value of 68.60 ± 0.91% .The increase in the mean value could be because of the increased intensity (1600 mW/cm^2) of the newer generation LED. The mean degree of polymerization was evaluated for Groups A, B and C for the thickness of 1mm. The subgroup 1mm of Group A showed percentage degree of polymerization of 61.53 ± 1.88% with a maximum value of 62.57 and minimum of 60.48. The subgroup 1mm of Group B showed percentage degree of polymerization of 64.40 ± 1.12% with a maximum value of 63.02 and minimum of 61.77. The subgroup 1mm of Group C showed percentage degree of polymerization of 55.66 ± 1.23% with a maximum value of 56.35 and minimum of 54.98.

Comparing the mean percentage degree of polymerization, Group B had the highest value of 64.40 ± 1.12%. The reason being, the intensity of the conventional LED was higher than that of the halogen though the curing time was less. When compared to the newer generation LED, the curing time was three times more, which gave better conversion results even though the intensity was less.The mean degree of polymerization was evaluated for group A, B and C for the thickness of 1.5mm.Group A subgroup 1.5mm showed percentage degree of polymerization of 56.33 ± 1.17% with a maximum value of 56.98 and minimum of 55.68.Group B subgroup 1.5mm showed percentage degree of polymerization of 47.2 ± 0.94% with a maximum value of 47.72 and minimum of 46.67.Group C subgroup 1.5mm showed percentage degree of polymerization of 48.73 ± 1.03% with a maximum value of 49.30 and minimum of 48.16.

Comparing the mean percentage degree of polymerization, Group A has the highest value of (56.33 ± 1.17%).Group B and C showed low values when compared to group A. The curing regimen with the halogen light was 40 seconds but the curing regimen for LED and Newer generation LED was only 20 seconds and 3 seconds respectively. The decrease in mean percentage degree of polymerization between the group A and Group B, C could be because of the difference in the curing times of the curing lights.

When we compare the percentage degree of polymerization within groups (i.e), the difference in % degree of conversion between the varying thicknesses (0.5mm, 1mm and 1.5mm) cured with their respective lights.The mean percentage degree of polymerization values of subgroup 0.5mm of all the Groups A, B and C were higher than the 1mm subgroups and their mean values were higher than the 1.5mm mean values of all the groups owing to the reduced thickness of composite resin.0.5mm mean values > 1mm mean values > 1.5mm mean values. An increase in resin composite thickness diminished the mean degree of conversion. When the sample thickness was 0.5mm the percentage degree of polymerization was greatest with the newer generation LED owing to its high intensity.As the sample thickness was increased, conventional LED and the Halogen when used showed increased percentage degree of polymerization compared to the newer generation LED. This could be because of the increase in curing time using the Halogen (40sec) and the conventional LED (20 sec) when compared to the newer generation LED (3 sec).

The null hypothesis stands rejected.

Further studies need to be done to testify whether the degree of polymerization will improve if the curing time is increased with the increase in thickness of composite when used with the high intensity newer generation LED.

VI. Conclusions

The degree of polymerization values decreased as the thickness increased.
Intensity of light has very little role in polymerization as the thickness increased.
Duration of cure proved to be more efficient in polymerization as the thickness of composite increased.
Hence, in clinical scenarios when lingual brackets need to be offset, the duration of cure must be increased sufficiently to achieve better outcome.

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