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Analyzing the impact of various curing techniques on the degree of conversion of various bulk fill composites - An in vitro study

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Abstract

Aims: To evaluate the effect of different light curing protocols: conventional, soft start and pulse delay on degree of conversion of bulk fill composites.

Methods and Material: Plastic mold of size 5mm diameter and 4mm height were used for the preparation of the study sample of 108.Three bulk-fill resin composites (Tetric N-Ceram bulk-fill, Filtek bulk fill, Xtra fill, and Filtek flowable) were compared in terms of degree of conversion. Three curing methods were used: 1) conventional, 2) soft-start curing and 3) Pulse delay. 36 specimens of each bulk-fill composite were fabricated and was subjected to Fourier transform infrared spectroscopy for measurement of absorbance peaks at 1608 and 1636.

Statistical analysis used: Data was analysed using SPSS version 23. Friedman's test for intra-group comparison and one-way ANOVA with post hoc tukey test for intergroup comparison.

Results: there is a statistically significant difference present in degree of conversion of all three composites:1) Tetric N-Ceram, 2) Filtek bulk fill and 3) X-trafil with various curing protocol: Conventional curing, soft start and Pulse delay.

Conclusions: The DC is affected by composition of the composite material and significant differences were found among various composites.

Keywords*:* Conventional Curing, Degree of Conversion, Fourier Transform Infrared Spectroscopy, Pulse Delay, Soft Start.

Introduction

Composites are well known for their aesthetics attributes, excellent mechanical properties, and minimal cytotoxicity. They are now material of choice for direct and indirect restorations on posterior and anterior teeth. Composite consist of various monomers which forms into a complex polymer structure on polymerization. The conversion of monomeric carbon-carbon double bonds into polymeric carbon–carbon single bonds is termed 'degree of conversion'. This Monomer conversion into polymers is never complete and so results in monomers that remain unreacted. The maximum conversion that is achieved is 75-85%. [1]

For achieving long-term durability of dental composites, it is important that most of the monomers convert into polymers during polymerization reaction. [2]There are many different kinds of variables that may influence the DC i.e curing light used, irradiation time, wavelength, power density, light-tip size, photo-activation method, amount of filler and their distribution in matrix, the form and amount of the photo-initiator, and shade of the composite resins.

The degree of conversion obtained during polymerization has a direct impact on the physical and mechanical characteristics of dental composites.[3]

Lower degree of conversion leads to inferior mechanical properties and greater discoloration and degradation over a period of time. As a result, such restorations have poor wear resistance and poor colour stability [4] A new generation of composites has been introduced, known as, Bulk-Fill Composite", which are supposed to achieve better physical and mechanical properties. They can be used in 4 to 5-mm thick increments using a mono-block or single layer technique.[5] The process of curing of composite resins occurs in three main phases: pre-gel, gel point and post-gel. [6] The polymerization continues even after curing and the maximum conversion is achieved till after half an hour of curing (though it is never complete). [7]

Thus, this study was carried out to evaluate the degree of conversion of various bulk-fill composites using different curing protocols.

Materials and Methodology

Circular Plastic molds of size 5mm diameter and 4mm height were used for the preparation of the study sample of 108.Three following bulk-fill resin composites were evaluated:

1. Tetric n-Ceram group
2. Filtek posterior bulkfill composite group
3. X-trafil group

Based on the composite type following groups and subgroups were studied:

GROUP 1-Tetric N-Ceram composite group (N=36)

Subgroup 1a- conventional curing protocol (n=12)

Subgroup 1b- soft start curing protocol (n=12)

Subgroup 1c- pulse delay curing protocol (n=12)

GROUP 2-Filtek bulk-fill posterior restorative composite group (N=36)

Subgroup 2a- conventional curing protocol (n=12)

Subgroup 2b soft start curing protocol (n=12)

Subgroup 2c- pulse delay curing protocol (n=12)

GROUP 3-X-tra fill composite group (N=36)

Subgroup 3a- conventional curing protocol (n=12)

Subgroup 3b- soft start curing protocol (n=12)

Subgroup 3c- pulse delay curing protocol (n=12)

Uncured composite was condensed into the mold and was subjected to FTIR for measurement of absorbance peaks at 1608 and 1636.For both cured and uncured composites absorbance peaks were recorded at 1608 and 1636.The prepared samples were covered with glass cover slip to remove excess material and light cured by using Polywave LED curing unit (Bluephase G2).

Three different curing protocols were followed: 1) Conventional photo-activation-An irradiance of 1200 mw/cm² for 30s

A. Soft start method -Light-curing was initiated with an irradiance of 650 mw/cm² for 5s,then was followed with an irradiance of 1200mw/cm² for 25s.
B. Pulse delay method –Initiated with 650 mw/cm² for 15s delay period of 3 minutes followed by irradiance of 650 mw/cm² for 15sec DC was evaluated immediately by FTIR spectrometer using attenuated total reflectance (ATR) technique. The DC was calculated using the formula below: [9]

DC % = [1 – 1636 cm-1/1608 cm-1) peak height after curing] * 100/1636 cm-1/1608 cm-1) peak height before curing

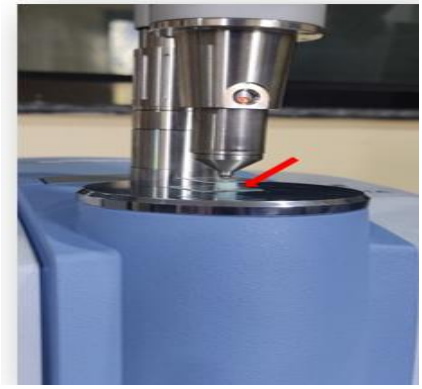

Figure 1 :Mounted sample on FTIR

Result

Intra-Group comparisons of group 1(1a, 1b 1c)showed mean values of 57.29, 55.29, 64.23,group 2 (2a, 2b 2c) showed mean values of 53.67,54.59,52.20 respectively, which were not significant.(P > 0.05). Group 3 (3a, 3b 3c) showed mean values of 54.11, 49.78, 52.59 respectively, which is significant. (P< 0.05) (Table 1). Sub-groups 1b,2b,3b and 1c,2c,3c show statistically higher degree of conversion. The DC of these sub groups -1a, 2a, 3a showed no significant difference. (Table2)

Table 1: Intergroup comparison of degree of conversion

Subgroup	N	Minimum	Maximum	Mean	Std. Deviation	Mean Rank	F Value	P Value
Conventional curing protocol								
Tetric N Ceram	12	42.22	57.71	50.97	5.56	42.22	1.26	0.327
Filtek	12	41.90	62.30	53.67	7.84	41.90		
Xtrafil	12	41.07	58.75	54.11	6.15	41.07		
Soft start curing protocol								
Tetric N Ceram	12	34.69	55.29	46.71	6.52	34.69	4.41	0.020
Filtek	12	40.63	64.29	54.59	7.35	40.63		
Xtrafil	12	39.93	58.08	49.78	5.79	39.93		
Pulse delay curing protocol								
Tetric N Ceram	12	40.17	64.23	50.17	8.15	40.17	2.33	0.047
Filtek	12	39.86	64.87	52.20	9.13	39.86		
Xtrafil	12	40.17	64.23	49.66	7.97	40.17		

Table 2: Intra group comparison of degree of conversion

Subgroup	N	Minimum	Maximum	Mean	Std. Deviation	Mean Rank	Friedman's Chi sq.	P Value
Tetric n ceram group								
Conventional	12	34.69	55.29	46.71	6.52	1.85	2.40	0.051
Soft Start	12	42.22	57.71	50.97	5.56	2.20		
Pulse Delay	12	40.17	64.23	49.66	7.97	2.00		
Filtek bulk fill group								
Conventional	12	40.63	64.29	54.59	7.35	2.25	1.50	0.472
Soft Start	12	41.90	62.30	53.67	7.84	2.00		
Pulse Delay	12	39.86	64.87	52.20	9.13	1.75		
X tra fill group								
Conventional	12	39.93	58.08	49.78	5.79	1.42	7.17	0.028
Soft Start	12	41.07	58.75	54.11	6.15	2.50		
Pulse delay	12	42.42	58.89	52.59	5.26	2.08		

Discussion

The term 'degree of conversion' refers to the conversion of monomeric carbon-carbon double bonds (c=c) into polymeric carbon-carbon single bonds (c-c). [10] higher the conversion, better is the physical and mechanical properties achieved.

In this study, prepared samples were mounted onto a diamond attenuated total reflectance (atr) accessory of a fourier transform infrared (ftir) spectrometer to evaluate dc. The ftir spectra were recorded with 16 scans, using a resolution of 2 cm-1 in the wavelength range of 600–2000 cm-1. [9]

a background ftir spectrum was collected before each measurement to remove background noise. Spectra for both cured and uncured composites were recorded under same conditions. Dc was calculated from the changes in the ratio of absorbance intensities (peak heights) of aliphatic c=c (1636 cm^{-1}) and aromatic c-c (1608 cm^{-1}) spectral bands. [9]

Intra-group comparison between -1a,1b,1c;2a,2b,2c shows p value of > 0.05 which is not significant while 3a,3b,3c shows p value of <0.05which is significant.

In our study, intergroup comparison shows-1a, 1b, and 1c maximum dc is with pulse delay method i.e, 64.23 and minimum is with conventional curing 34.69. For 2a,2b and 2c maximum dc is with pulse delay method is 64.87 and minimum is also with pulse delay 39.86. For 3a,3b, and 3c maximum dc is with pulse delay method is 58.89 and minimum is with conventional curing 39.93.

Comparison of various curing protocols i.e. Conventional curing, soft start and pulse delay methods, degree of conversion amongst subgroups was found to be-2a>3a>1a which was not statistically significant, while it was found to be-2b>3b>1b>2c>1c,3c which was statistically significant.

Among several methods to determine dc of composites, fourier transform infrared spectroscopy (ftir) has been widely used as a reliable method due to the availability of equipment and numerous sampling techniques and represents a simple convenient method whose results are consistent with the results of other more complicated techniques. This method detects the (c= c) stretching vibrations, centered around 1636 cm^{-1}& 1608 cm^{-1} directly before and after curing of materials. [9]

In the present study, plastic molds were used because it was easy and fast way to standardizing the samples. [11]

In the present study irrespective of the curing protocol used, filtek bulk-fill composite showed highest dc followed by x-tra fill composite. This may be due to presence of udma in the formulation of filtek bulk fill posterior composite and which may have resulted in higher dc which was in accordance with the study done by a. Nour a. Habib, gihan h. Waly. [12]the viscosity of udma is much lower and flexibility is higher compare to bis-gma due to weak hydrogen bond. Micro structure of udma monomer is responsible increased mobility of radical sites on the polymer network and consequently enhanced polymerization and monomer conversion. [13]

Higher dc of x-tra fill in our study may be due to its organic matrix, which is composed of bis-gma, udma, and tegdma. Sideridou et al showed that the maximum dc of different monomer systems increases in the following order: tegdma>udma>bisema>bis-gma. [14,15]

The lowest dc in tetric n -ceram may be related to low penetration of light into the composite which is similar to the results of study done by samaneh rezaei. [14]

As mentioned in the study done by magali dewaele et al, the volumetric shrinkage of composites has been shown to be proportional to its degree of conversion. It has been found that an ideal composite exhibits a minimal polymerization shrinkage with an optimal degree of conversion. As an increase of monomer conversion leads to the increase of polymerization shrinkage, these seem to be antagonistic. [16]

Dc depends on factors like curing time, matrix composition, light intensity, filler content, diluent concentration and initiator concentration, has a large role in determining the physical and mechanical properties of the material such as hardness, tensile strength, compressive strength, wear resistance etc. [17]

Camargo ej et al and stanbury et al did a study in which they measured shrinkage stress and conversion simultaneously and also provided evidence that the increase in filler content results in slight reduction of dc. Low molecular weight monomers reduce the viscosity of resin composites and enhance dc but they increase thereby polymerization shrinkage stress. [17]

Ajaj ra and gonclave et al based on their study stated that dc tests with different materials regardless of the curing mode showed that there is a statistically significant difference between materials which is similar to our study. This may be due to differences in micro structural composition of the material like filler shape and size, their distribution in continuous phase i.e resin matrix, this affects light distribution and subsequently polymerization of the material. [18,19]

Both the type of material and the curing mode were found to be factors that have a statistically significant effect on the dc of the resin composite. Ceballos et al. Reported depth of cure using the scraping technique and the micro hardness using the calibrated vickers indenter: they concluded that the type of light-curing unit affected the curing effectiveness, and that the duration of exposure, the composite brand, and its thickness also had great effects. [19]

Conclusion

There were significant differences in dc between various composites. Irrespective of the curing protocol used, filtek group showed highest dc. Dc is more influenced by composition of composite material than the curing protocol used, though dc of composite was found to be higher when cured with pulse delay method as compare to soft start and conventional curing method.

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