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Evaluation of effect of different curing protocols on polymerization shrinkage stress of various bulk fill composites-An in vitro study

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Abstract

The current study is aimed to evaluate the effect of three different curing protocols i.e. conventional curing, soft start and pulse delay curing on shrinkage stress of various bulk fill composites. Three bulk-fill resin composites (Tetric N-Ceram bulk-fill, Xtra fill, and Filtek Bulkfill) with the sample size of 108 were compared for polymerization shrinkage stress. Curing methods used were 1) conventional, 2) soft-start curing and 3) Pulse delay. 36 disk-shaped specimens of each composite with a dimension of 1 mm thickness, 4 mm breadth and 10mm length were fabricated. Universal testing machine (UTM) with a speed of 1mm/min was used to measure the shrinkage stress generated during polymerization by load cell (50 kg) attachment. Movement of load cell was continuously monitored and recorded using software (blue hill) Data was recorded as force (in Newton)* Time (in seconds) and converted to MPa by dividing the force by the areas of transverse section of specimens (4 mm^2). 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. Statistically significant difference was present in PSS of all three composites with highest PSS when cured with conventional curing method, lesser with soft start and least in pulse delay curing method. Irrespective of the bulk fill composites used the soft start and pulse delay curing protocols cause less PSS and seems to be promising curing methods. Less PSS will lead to less stress at tooth-restoration interface and probably prevent debonding.

Keywords: Bulk-fill composite, Polymerization shrinkage stress, Pulse delay curing, soft start curing, Universal Testing machine.

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1. Introduction

Resin-based composite (RBC) restorations are popular, as they are esthetic restorations and have good durability due to advances in their compositional micro-structure [1,2]. Monomeric resins convert into polymers by exposure to visible light. The polymerization is usually incomplete, maximum achievable conversion with present day composites is 75-85%, leaving a significant proportion of methacrylate groups unreacted [3]. The residual unreacted monomer acts as a plasticizer and reduces the mechanical properties of the restorative material. They can also produce allergic reactions & discoloured restorations [4]. Inherent shortcoming of composite is their volumetric shrinkage due to polymerization, resulting in polymerization shrinkage stress, which is defined as shrinkage that takes place in such a way that the restoration material is being pulled away from the cavity walls' resulting in gap formation, stress generation between the restoration and the cavity wall during the polymerization sequence. This reaction called the gel effect or Trommsdorf-Norrish effect is self-limiting, which arises from the increase in system viscosity resulting in decrease in mobility of the reactive species and is imposed by the rapid formation of a highly cross-linked polymeric network. This stress is transmitted to the tooth–restoration interface and may lead to gap formation, micro-leakage, marginal breakdown, enamel cracks and cuspal deflection. This could lead to secondary caries and/or restoration loss [4,5]. The clinician strives to achieve maximum conversion of monomers to polymer by adopting methods like- selecting appropriate curing light, using different light curing protocols, highly filled composites etc, so as to achieve optimal physical and mechanical properties which, in turn will reduce the number of un-reacted methacrylate groups and maximise the longetivity of restoration. Curing has three phases i.e. pre gel, post gel and gel point [6 ,7]. In pre-gel phase, the material has ability to flow and undergo molecular rearrangement, to compensate for shrinkage. This phase is marked by a linear polymer chains. As the polymerization progresses the resin becomes more viscous (post gel), reaching its gel point [6,7]. Before the gel-point is reached, composite vitrification can be extended using various curing protocols to allow for the composite to flow, to reduce the shrinkage stress. As the composite becomes more viscous, its flow reduces and transmits the stress caused by polymerization shrinkage at the interface. At this stage, there is a predominance of cross links in the polymer structure [6 ,7]. Polymerization shrinkage still poses a critical challenge to clinicians in composite restorations as it can lead to internal stresses as well as stresses at the margins of restoration which over time may result in the formation of micro-gaps and cohesive and adhesive failure between the restoration and internal cavity walls and lead to a "micro-leakage sequel" marginal staining, postoperative sensitivity, secondary caries. In addition, shrinkage stress can propagate micro-cracks within the restorative material [8]. One of the methods to lessen polymerization shrinkage stress is use of highly filled composites. Bulk-fill composites perform well in terms of degree of conversion and polymerization shrinkage in spite of being used in 4-5 mm thickness. Another method to combat the shrinkage stresses is using different curing protocols.9Thus, the aim of the study was to evaluate the effect of three different curing protocols- Conventional, Soft start, Pulse delay curing methods on shrinkage behaviour of various bulk fill packable composites.

Null Hypothesis: There is no difference in Polymerization shrinkage stress of composite when polymerized with different curing protocols.

2. Materials and methods

To measure the shrinkage stress 108 samples were prepared using three different bulk fill composites and each type of composite was cured by three different curing protocols and grouped as below:

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)

The samples were prepared by placing composite between 2 custom-made glass jigs. The glass jigs were 4mm in width and marked for 10 mm of length of the glass jig. Silane coupling agent (Ultradent) was applied on the facing side of each glass jigs followed by application of a thin layer of self- etch adhesive and light cured for 20 sec. To achieve vertical gap of 1mm both prepared surface of glass jigs was placed against each- other and fixed on measuring machine, followed by placement of bulk-fill composites between the prepared surface of glass jigs to form composite specimen of 1 mm thickness, 4 mm breadth and 10 mm length. All the samples of each group were then subjected to photo-polymerization using three curing protocols by polywave curing unit (Bluephase G2Polywave, IvoclarVivadent):

a) Conventional photo-activation-An irradiance of 1200mW/cm² for 30s

b)Soft start method-Light-curing was initiated with an irradiance of 650mW/cm² for 5s, then was followed with an irradiance of 1200mW/cm² for 25s.

c) Pulse delay method–Initiated with 650mW/cm² for 15s delay period for 3 minutes followed by irradiance of 650mW/cm² for 15 sec

UTM was used with a speed of 1mm/min to measure the shrinkage stress generated during polymerization by load cell (50 kg) attached to the curing composite which caused slight movement of load cell that was continuously monitored and recorded using software (blue hill) Data was recorded as force (in Newton)* Time (in seconds) in graphs and converted to MPa by dividing the force by the areas of transverse section of specimens (4 mm^2).

3. Results

Data was analysed using SPSS version 23. Descriptive statistics, Friedman's test for intra-group comparison and one-way ANOVA with post hoc tukey test was done for intergroup comparison. Intra-Group comparison- 1a, 1b and 1c showed a mean value of 10.86, 5.96, 5.08 respectively, 2a, 2b and 2c showed a mean value of 11.23,6.03,4.78 respectively, 3a, 3b and 3c showed a mean value of 11.00, 5.68, 5.10 respectively with a P value of < 0.001 which were highly significant (Table 2). Intergroup comparison with various curing strategies was not statistically significant (Table1). However statistically significant differences were present in PSS of all three composites when cured with different curing protocols. Groups 1(1a,1b,1c), 2 (2a,2b,2c) and 3 (3a,3b,3c) showed statistically significant difference in PSS which was highest in conventional curing method, lesser in soft start cure and least in pulse delay curing method.

4. Discussions

A key goal in the research and development of composites is to improve their clinical durability and ease of use. Over the years lots of advances have taken place to improve the mechanical properties and decrease or compensate for the PSS of the composites like improvements in filler, matrix and initiator technology [2]. According to the vast majority of published literature, bulk-fill composite performs well in terms of polymerization shrinkage [9]. Third-generation LED, also called ''Poly-wave,'' provide a broad-spectrum light-curing unit that can activate all current photo-initiators present in composites. Such as Lucirin TPO and derivatives of dibenzoyl germanium such as Ivocerin [10]. For maximum conversion a sufficient amount of light energy of particular wave-length should be irradiated. Two modified light curing protocols, soft start and pulse-delayed, were introduced as an alternative to the standard protocol [11]. Soft start involves two step curing method. Initially, lower light intensity is applied in order to extend the gel phase. In second phase, light intensity increases exponentially to its maximum value during a 10-second period and remains constant for the duration of exposure for 30 sec [12,13]. The pulse-delayed protocol provides a relaxation interval (dark interval) between the first pulse and subsequent pulse. This dark interval gives material, time for relaxation, allowing it to flow, thereby reducing stress.

Table 1: Overall intergroup comparison of polymerization shrinkage stress

Subgroup	N	Minimum	Maximum	Mean	Std. Deviation	Mean Rank	F Value	P Value
Conventional curing protocol								
Tetric N Ceram	12	10.12	11.98	10.86	0.78	10.12	0.933	0.404
Filtek	12	10.35	11.92	11.23	0.45	10.35		
Xtrafil	12	10.01	11.93	11.00	0.76	10.01		
Soft start curing protocol								
Tetric N Ceram	12	5.03	6.94	5.96	0.69	5.03	1.15	0.329
Filtek	12	5.11	6.93	6.03	0.65	5.11		
Xtrafil	12	5.13	6.32	5.68	0.46	5.13		
Pulse delay curing protocol								
Tetric N Ceram	12	4.48	5.88	5.08	0.42	4.48	1.62	0.213
Filtek	12	4.02	5.61	4.78	0.53	4.02		
Xtrafil	12	4.02	5.91	5.10	0.52	4.02		

Table 2: Intra group comparison of polymerization shrinkage stress

Subgroup	N	Minimum	Maximum	Mean	Std. Deviation	Mean Rank	Friedman's Chi sq.	P Value
Tetric N cerum composite								
Conventional	12	10.12	11.98	10.86	0.78	3.00	19.5	<0.001
Soft Start	12	5.03	6.94	5.96	0.69	1.75		
Pulse Delay	12	4.48	5.88	5.08	0.42	1.25		
Filtek Bulk fill composite								
Conventional	12	10.35	11.92	11.23	0.45	3.00	22.167	<0.001
Soft Start	12	5.11	6.93	6.03	0.65	1.92		
Pulse Delay	12	4.02	5.61	4.78	0.53	1.08		
X tra fill bulk fill composite								
Conventional	12	10.01	11.93	11.00	0.76	3.00	19.5	<0.001
Soft Start	12	5.13	6.32	5.68	0.46	1.75		
Pulse Delay	12	4.02	5.91	5.10	0.52	1.25		

During this stage, stress reduction also takes place due to formation of a more linear structure initially with less cross-linking between the polymer chains. The dark interval duration still remains a topic of researches for many [14]. The polymerization shrinkage has an inverse correlation with filler content. So, composites having higher filler content have less polymerization shrinkage [15,16]. In our study the glass jig surfaces were silanized to improve bonding. The results of present study showed that PSS varied among different curing protocols of same composite. Mean values for Tetric N-ceram group with various curing protocols i.e. conventional -1a, soft start-1b and pulse delay-1c method is 10.86, 5.96, 5.08 MPa respectively. (Table 1). For Filtek group with conventional -2a, soft start -2b and pulse delay -2c method is 11.23, 6.03, 4.78 MPa respectively. (Table 1). For X-tra fill group with conventional -3a, soft start -3b and pulse delay-3c method is 11.0, 5.68, 5.10 MPa respectively. (Table 1). Intra group Comparison of each group -1a,1b,1c; 2a,2b,2c; 3a,3b,3c shows statistically highly significant difference. The PSS amongst all the sub-groups was found to be :1a>3a>2a>1b>2b>3b>3c>1c>2c(Table no.1). In our study PSS of Bulk-fill fill composites (Tetric n-ceram, filtek bulk fill, x-tra fill) was higher when cured with conventional curing protocol and least with pulse delay protocol so the null hypothesis was rejected. Cunha et al used Modulated curing methods and showed effective reduction of rate of shrinkage stress which improved bond strength at interface [17]. Study done by Fabio Antonio Piola RIZZANTE who found that Bulk-fill composites showed equal or lower PSS when compared to conventional composites, especially when thicker increments were evaluated.18 HM El-Damanhoury et al also found that bulk-fill composites resulted in significant reduction in PSS while maintaining adequate curing at a 4-mm thickness [19]. In our study PSS was found to be least in pulse delay and soft start curing methods which is in accordance with the study done by Bomfim et al which claimed that slower polymerization causes an improved flow of molecules in material, decreasing PSS in restoration. During the polymerization process, thermal variation (developed temperature) may increase stresses into the material if no time is allowed to dissipate this thermal energy. So, stresses generated with initial low irradiance will be less, due to less alteration of temperature [20]. Another important parameter is C-factor (configuration factor) defined as the ratio between bonded and un-bonded surfaces [21]. The application of the C-factor concept to clinical practice is a much more complex geometry than the specimens used in in-vitro mechanical testing, resulting in a very heterogeneous stress distribution.

The correlation between C-factor and magnitude of shrinkage stress is highly dependent on bonded substrate. This correlation can be considered to be responsible for numerous inconsistent results reported in the literature as mentioned in the study done by Zhengzhi Wang et al [22]. Shrinkage stress measurements by mechanical testing present limitations. Such as, 1) shrinking composite develops a tri-axial stress state, while stress manifested only in the long axis is registered. 2) Stiffness of the bonding substrate leads to low compliance of the testing system [21]. Modifying the light-activation protocol based on the concept of delaying the composite vitrification to allow relief of shrinkage stress by prolonging the period that composite can flow, has been advocated to reduce shrinkage stress [23]. Several laboratory studies have demonstrated improvements in marginal integrity of restorations using these protocols to modify light-activation without compromising mechanical properties of composites [23]. However, these modified light-activation protocols may have limitations. Using low irradiance generates few chain growth centers, resulting more linear polymeric chains; which are more prone to degradation. Moreover, slower polymerization reaction might produce polymers with lower elastic modulus than those obtained under high irradiance, contributing to reduction in shrinkage stress [23]. In our study we found that PSS was higher in conventional curing mode, as compared to soft start and pulse delay modes. Similar results were found by a study done by Lu H et al who used an experimental set-up that allowed real-time measurement of shrinkage stress and degree of conversion within same specimen, to compare light-activation with the soft-start, pulse-delay and continuous modes [24]. There is no consensus in the literature about the benefits of different light application protocols and little clinical data is available to show if such protocols provide significant benefits under clinical conditions. The variability in polymerization shrinkage stress may be related to the differences in matrix formulation, filler content, type and shape of cavity configuration.

5. Conclusions

Based on the results of the present in-vitro study, following can be concluded:

1) Statistically significant differences were found in the PSS of all three composites i.e. Tetric N-ceram, Filtek bulkfill and X-tra fil when cured with different curing protocols i.e. conventional curing, soft start and pulse delay.
2) All groups showed higher PSS when cured with conventional curing method and least with pulse delay method.
3) The soft start and pulse delay curing protocols cause less PSS and seems to be promising curing methods. As less PSS will lead to less stress at tooth-restoration interface, probably resulting in decrease in bond failure and increase in longetivity of restorations.

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