Thermal Stability and Monomer Elution of Bulk Fill Composite Resins Cured from Different Irradiation Distances

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Article Info	A B S T R A C T
Article type: Original Article	**Objectives:** This study aimed to evaluate the thermal stability and monomer elution of bulk fill composite resins cured at different irradiation distances.
	Materials and Methods: Forty cylindrical-shaped (3×4mm) specimens were fabricated from two composite resins (X-tra fil, X-tra base) and cured from 0 and 7mm distances. In 9 specimens, the degree of conversion was determined by the release of monomers. For this purpose, after curing of composites, they were immersed in 5 mL of 99.9% methanol and stored at 37°C for 24h. The eluted monomer was then analyzed by gas chromatography (GC). Also, thermal stability of one sample from each group was assessed by thermogravimetric analysis (TGA) at a rate of 10°C/min. Data were analyzed using two-way ANOVA and Tukey's post-hoc test (P<0.05).
* Corresponding author: Department of Operative Dentistry, Dental Faculty, Tabriz University of Medical Sciences, Tabriz, Iran Email: pournaghiazarf@gmail.com	**Results:** X-tra fil had significantly higher degree of conversion than X-tra base (P=0.001). Specimens cured at 7mm distance had significantly lower degree of conversion compared with those cured at 0 mm distance (P=0.001). The interaction effect of composite type and distance of light curing unit from the surface of samples was statistically significant (P=0.001). Regarding the TGA results, the lowest and the highest percentages of weight loss were detected in X-tra fil cured at 0 mm distance and X-tra base cured at 7mm distance, respectively.
	Conclusion: X-tra fil composite cured at 0mm distance had the highest degree of conversion and thermal stability, and X-tra base composite cured at 7mm distance had the lowest values.

INTRODUCTION

Dental composites play an important role in modern restorative dentistry. Durability and efficacy of these materials are influenced by their chemomechanical properties as well as the experience and expertise of dental clinicians. The layering technique is a common method of application of conventional composites in the clinical setting. Composite is applied in maximum thickness of 2mm for each curing cycle in order to decrease the polymerization shrinkage and subsequent stress. This technique is common due to its high clinical success rate; however, it has several limitations. It is time consuming and has high technical complexity due to limited curing

depth, risk of air bubble retention, and risk of contamination between the layers, which would lead to detachment of layers, and difficulty in restoring small cavities [1-5].

Recently, the tendency to use bulk fill composites has increased to overcome the problems associated with the layering technique. In this technique, bulk filling of the cavities with composite applied in 4-5 mm or greater thicknesses has been proposed, yielding lower shrinkage stress during polymerization and deeper curing [6]. Bulk fill composites are produced in two viscosities of low (flowable) and medium (paste-like). Both types have lower amounts of fillers with larger size and higher translucency to achieve high curing depth following increased light transmission [2,7]. Some certain photo-initiators may be used in some of these composites to provide higher depth of curing. Also, monomers with lower double-bond concentration may be used [8]. Despite all the above, significantly lower depth of cure has been reported for some bulk fill composites compared with the value claimed by the manufacturers [9].

Polymerization of light-activated materials is influenced by several factors, such as the exposure time, light intensity, and distance between the tip of the light curing unit and the surface of the restorative material [4]. The distance between the tip of the light curing unit and the restoration surface varies depending on the depth of the cavity, and may cause changes in the rate of polymerization. the larger the distance, the lower the intensity of light would be. Preferably, the tip of the light curing unit should be in contact with the composite surface; although, it may not be feasible in most cases considering the shape of the cavity [10]. This is especially important in bulk fill composites due to their higher thickness. Insufficient polymerization is a major drawback of all composite resins. In clinical situations, not all dimethacrylate monomers are converted to polymers, and a significant amount of monomers remain unreacted, forming the main part of the resin matrix (20% to 40% by weight). Unreacted monomers can lead to bio-toxic effects and weaken the mechanical properties of the

material by leaving the material mass. Increasing the cross-link density reduces the empty volume and polymer network porosity through the proximity of polymer chains. This provides a limited elution path for residual monomers to propagate outside the structure [11].

One method for assessing the degree of crosslinking density is to assess the thermal stability under the initial degradation temperature measured by thermogravimetric analysis (TGA). The TGA of dental composite resins investigates the percentage by weight of inorganic filler content, the initial degradation temperature, and the thermal degradation kinetics [2,12]. Moreover, the quality and quantity of the residual monomers eluted from the polymerized dental composites can be determined by gas chromatography (GC), which is used to indirectly investigate the level of curing and degree of conversion [13-16].

In order to study the effect of distance from the light curing unit tip on the polymerization rate, we measured the amount of monomer elution from two bulk fill composites one day after polymerization as well as their thermal stability. Since in the clinical setting, the distance of the light curing unit tip from the surface of the composite restoration is at most 7mm, the 0 and 7mm distances were evaluated in the present study [17,18]. The null hypothesis of this study was that the rate of monomer elution and thermal stability of the two types of bulk fill composites cured at 0 and 7mm distances from the light curing unit would be the same one day after polymerization.

MATERIALS AND METHODS

In this experimental study, 10 samples were fabricated in each group; of which, 9 and 1 were used for the evaluation of monomer elution and thermal stability analysis, respectively. Cylindrical composite samples with 3 mm diameter and 4 mm height were fabricated using a Teflon mold. For this purpose, the mold was placed on a glass slab and fully filled with composite. Then, the samples were cured by a LED curing unit (Dentamerica 686, DENTAMERICA Inc., CA, USA) at 0 and 7mm distances after placing a thin celluloid strip over them.

Table 1. General characteristics of the composite resins evaluated in this study (maximum layer thickness is 4mm for both materials)

Material	Capping layer	Organic matrix	Filler (wt%/vol%)	Shade
X-tra fil (Voco; Cuxhaven, Germany)	Not required	Bis-GMA, UDMA, TEGDMA	86/70	Universal
X-tra base (Voco; Cuxhaven, Germany)	Required	Bis-EMA, aliphatic dimethacrylate	75/61	A2

Bis-GMA: bisphenol A-glycidyl methacrylate; UDMA: urethane dimethacrylate; TEGDMA: triethylene glycol dimethacrylate; Bis-EMA: bisphenol A ethoxylate dimethacrylate

The light intensity was measured by a light meter, which was found to be approximately 800mW/cm^2. According to the manufacturer's instructions, the curing time was considered to be 20 s. The characteristics of the bulk fill composites used are presented in Table 1. For GC, after curing of the samples under the defined conditions, the specimens were stored in brown glass containers containing 5 mL of 99.9% methanol in a dark place at 37°C for 24 h, and then a Varian Star 3400 GC machine equipped with silica-packed column (VarianSaturn3; Agilent Technology Inc., Santa Clara, USA) was utilized to measure the level of monomer elution. The injection valve temperature was set at 260°C, and the temperature began to rise at a rate of 3°/min. The temperature of the device remained at 40°C and 100 °C for 1 and 2 min, respectively. The peak generated at the 1.59-min moment was related to methyl methacrylate. Examining the integral below the graph indicated the monomer elution level, which was reported to be different for various samples with different curing conditions [16]. TGA was performed using the TGA machine (Linseis STA PT 1000; Linseis Inc., NJ, USA). Also, 0.05 g composite samples from each group were fabricated and placed in the device furnace after weighing. The analyzer was set at 10°C/min temperature increase rate in the air. The weight loss percentage was reported for various samples with different curing conditions according to the weight loss graph in terms of temperature [19].

Statistical analysis:

Data of GC for monomer elution were reported using descriptive statistics of mean ± standard deviation.

To analyze the variables, two-way ANOVA was applied in SPSS version 17.0 (SPSS Inc., Chicago, IL, USA). The Tukey's post-hoc test was used for pairwise comparisons of the groups. Subgroup analysis was performed using one-way ANOVA. The P values were corrected for multiple comparisons by the Bonferroni test. The significance level was set at $P<0.05$.

RESULTS

Regarding the GC results, descriptive statistics of the monomer elution of the specimens (in mM) are presented in Table 2.

Table 2. Mean and standard deviation of monomer elusion (mM)

Composite resin	Light curing distance (mm)	
	0	7
X-tra fil	5.75±0.05	20.13±0.33
X-tra base	15.55±0.40	26.83±0.95

The lowest and the highest monomer elution rates were obtained for X-tra fil composite at 0 mm distance from the light curing unit and X-tra base composite with 7 mm distance, respectively. Two-way ANOVA was employed considering the presence of two variables of composite type and distance. Based on the results, the effect of composite type was statistically significant (P=0.001).

The effect of distance between the tip of the light curing unit and the surface of the sample was also significant (P=0.001). There was a significant interaction between the composite type and the distance between the light curing unit and the surface of the samples (P=0.001).

Subgroup analysis was performed to find within group differences. The results revealed a significant difference in the mean values of monomer elution between X-tra fil/0-mm distance and X-tra fil/7-mm distance groups (P<0.001). Moreover, monomer elution in X-tra base/0-mm and 7-mm distance groups was significantly different (P<0.001). In both composite groups, monomer elution increased in 7 mm distance with greater increase in X-tra fil group. Also, between-group comparisons showed that the mean values of monomer elution were significantly different in X-tra fil and X-tra base for both distances. The mean values of monomer elution in different groups are depicted in Figures 1 and 2.
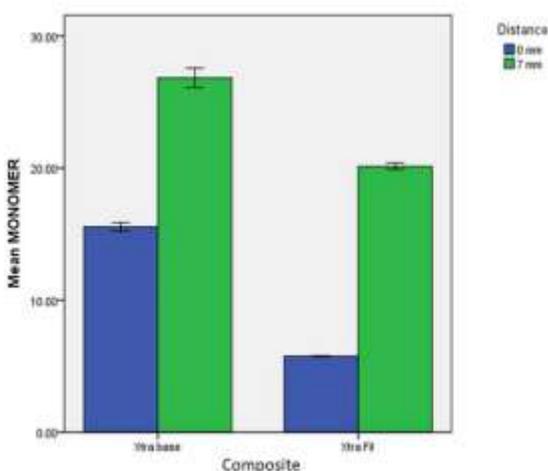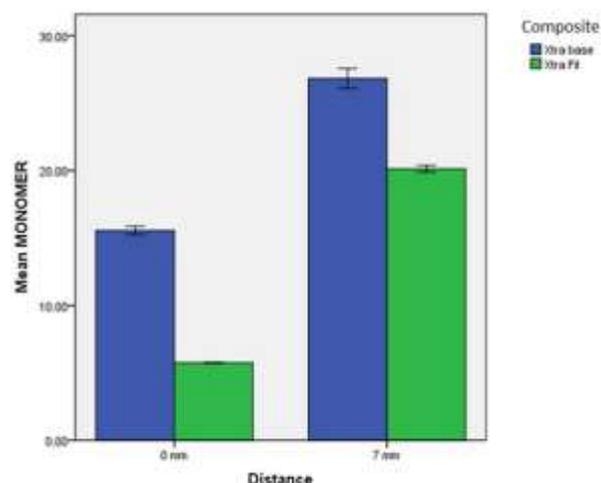

Fig. 1: Mean amount of eluted monomer (mM) based on the type of composite at different curing distances (mm)

Regarding the TGA results, the lowest and the highest percentage of weight loss were noted in X-tra fil at 0 mm distance from the light curing unit and X-tra base at 7 mm distance, respectively (Table 3).

In the X-tra base composite groups, the monomer evaporation rate stood at 0.95 of the mass percentage at temperatures below 210°C (composite degradation temperature) at 0 mm curing distance and 1.93 of the mass percentage at 7 mm curing distance. The second drop in the TGA graph, associated with the degradation of the structure of the X-tra base composite, began at 210°C, which was lower than that of the X-tra

fil composite (345°C) (Fig. 3).

Fig. 2: Mean amount of eluted monomer (mM) based on the distance from the light curing unit tip (mm) in the two composite resins

Table 3. Weight loss of specimens according to TGA (%)

Composite resin	Light curing distance (mm)	
	0	7
X-tra fil	0.57	1.40
X-tra base	0.95	1.93

In the X-tra fil composite groups, the monomer evaporation rate was set at 0.57 of the mass percentage at temperatures below 345°C (composite degradation temperature) at the curing distance of 0 mm and 1.45 of the mass percentage at the curing distance of 7 mm.

The second drop in the TGA graph, which was related to the destruction of the X-tra fil composite structure, began at 345°C, which was higher than that of the X-tra base composite (210°C) (Fig. 3).

DISCUSSION

Proper polymerization of bulk fill composite resins is an important factor affecting their clinical success. Although the main advantage of these composites is their increased curing depth, there are some concerns regarding the rate of polymerization in higher thicknesses [20-22].
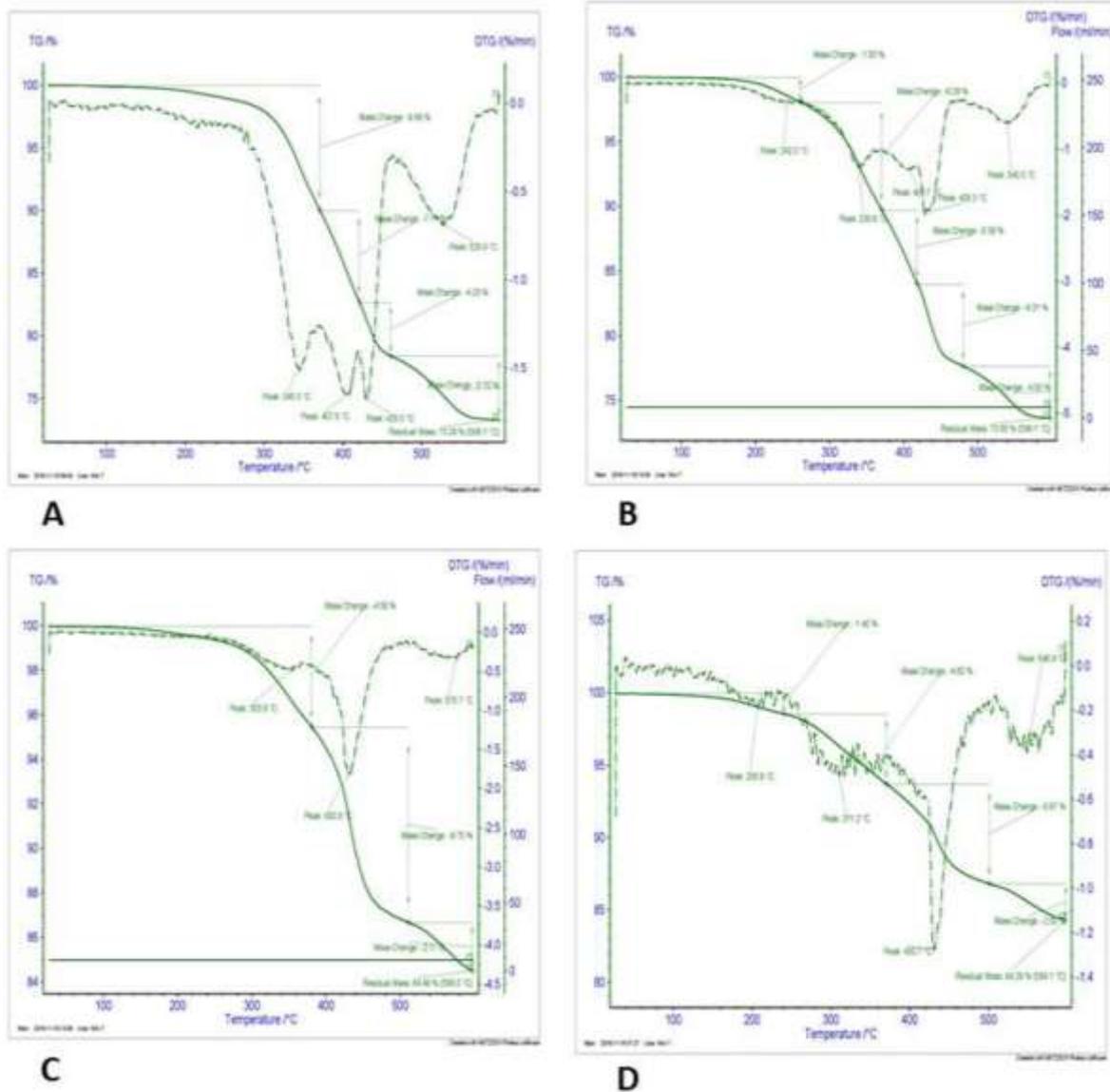
Fig. 3: TGA/DTGA diagrams: (A) X-tra base composite/0 mm distance; (B) X-tra base composite/7 mm distance; (C) X-tra fil composite/0 mm distance; (D) X-tra fil composite/7 mm distance

In the present study, GC was used to indirectly evaluate the polymerization rate of X-tra fil and X-tra base composites. By doing so, the effect of curing distance (0 and 7 mm) and composite type (flowable/ packable) on the degree of conversion of the composite samples during polymerization was investigated. According to the manufacturer's claims, an acceptable cure can be achieved in 4 mm thickness; thus, all specimens were fabricated with 4 mm thickness. In addition to the polymerization level, the degree of cross-link density of both composites was compared independently from the curing distance, by examining the thermal stability via TGA/DTGA graphs. The results of this study showed that the highest degree of conversion belonged to the bulk fill composite with higher viscosity (X-tra fil), cured at 0 mm distance. In other words, type of composite resin and the distance of the specimen from the light curing unit were critical factors in polymerization of specimens. Therefore, the null hypothesis was rejected. The degree of conversion of composite restorations can indicate their physical, mechanical, and biological

properties. Low conversion rates may increase the rate of elution of the non-reacted monomers, resulting in lower biocompatibility of the restoration.

In addition, uncured functional groups can act as a plasticizer and create a restorative material with lower mechanical properties [21]. Similar to our study, Aromaa et al. [23] concluded that with an increase in the distance between the tip of the light curing unit and the composite surface, the radiation absorbed by the composite and thereby the polymerization rate decreased. They reported that the optimal distance from the curing unit was 4 to 6 mm to achieve the most efficient ratio of radiation; although the most effective light transmission from the material was achieved in contact mode. In another study, Ilie et al. [24] indicated that the modulus of elasticity and degree of polymerization, independent of the type of composite, decreased with increasing distance from the curing unit or with increasing the thickness of the material, which could be due to lower energy quantum resulting in decreased polymerization activation [24]. This result is also consistent with the results obtained in our study, because, regardless of the composite type, in each composite group, samples that were cured from a longer distance showed lower degree of conversion and higher monomer elution.

According to the results of the present study, type of composite also affected the degree of polymerization. Based on the technical information provided by the manufacturer, X-tra fil is a paste-like hybrid radiopaque composite designed for use in the posterior region and contains 86wt% filler. It can be reliably cured in 4 mm layers, and its physical strength and resistance to abrasion will be high enough to withstand occlusal forces [25]. On the other hand, in X-tra base, which is a flowable bulk fill radiopaque composite, the cure depth increases compared with the conventional flowable composites and the shrinkage stress decreases. It can be cured in layers with 4 mm thickness, similar to X-tra fill; however, its inorganic filler rate is lower than that of the X-tra fil (75wt%) [26]. Various methods have been adopted to increase the depth of cure in bulk fill composites. An increase in the dimensions of fillers, and consequently, reduction of filler rate and the total filler surface, is an effective factor to enhance translucency. Increasing the size of fillers to 20 μm or more with the same filler content as seen in both studied composites decreases the total filler surface and the filler-matrix interface. Another factor in increasing the depth of cure is the similarity of the refractive index of the filler particles and the resin matrix, which results in light scattering within the material rather than at the filler-matrix interface. This allows for better penetration of light into the internal layers, and greater light transmission results in better curing depth for bulk fill composites [24, 27, 28]. In particular, LED curing units enable greater light penetration into deeper layers due to higher energy density [29]. On the other hand, X-tra base composite contains Bis-EMA, which has lower hydroxyl groups compared with Bis-GMA, and shows more flexibility due to its inability to form strong intermolecular hydrogen bonds, leading to higher degree of conversion [30, 31]. Another factor affecting the increase in polymerization rate of the bulk fill composites is the different photo-initiators compared with those of conventional composite resins. For example, Tetric Evoceram bulk fill composite has an enhancing initiator (Ivocerin) in addition to the usual camphorquinone/amine initiator system, which leads to higher polymerization depth. However, in X-tra fil and X-tra base, no changes have been made in the polymerization initiator system; thus, increased curing depth in these composites is the result of enhanced translucency [32].

According to the abovementioned statements and the manufacturer's claim, the differences observed in the polymerization rate and degree of conversion between the two bulk fill composites in the present study, independent of the distance of curing, could be related to their composition and their filler content. Higher filler content in X-tra fil than X-tra base could be a reason for its better polymerization rate and lower monomer elusion.

According to a study by Ilie et al, [32] filler volume had the highest effect on mechanical

properties, followed by filler weight, and the type of composite. X-tra base composite had a lower mechanical performance than X-tra fil composite. According to the manufacturer's recommendation, these composites require an additional capping layer of universal or posterior resin-based composites; however, X-tra fil composite can be used alone [7, 26]. Another study argued that the swelling behavior of some bulk fill composites is concerning, and the need for a capping material is not only for esthetic purposes, but it is also necessary to reduce the destructive effects on the material [31].

Pongprueksa et al. [6] evaluated monomer elution and degree of conversion of three types of composites from one manufacturer. Despite similar monomer content, the total monomer elution rate was significantly different depending on the composite type. The total monomer elution for a typical paste-like composite (Filtek Z250) was significantly lower than that of the universal (Filtek Supreme XTE) and bulk fill flowable (Filtek Bulk fill) composites. For the two flowable composites, the total monomer elution was significantly higher, regardless of the method of use (bulk fill or layering technique). As a result, the flowable bulk fill composite showed a significantly lower conversion rate at 4 mm depth (deepest area from the surface) and a higher total monomer elution, confirming the results of our study [6].

Accordingly, in our study, the results of the TGA, which measures the thermal stability of the composites, confirmed that the initial degradation of composite resin structure occurred at a higher temperature (345°C) in X-tra fil composite, compared with X-tra base (210°C), following a temperature rise of 10°C/min. The early weight loss below 210°, which is the composite degradation temperature, is due to monomer evaporation prior to decomposition of the composite, indicating a lower degree of polymer cross-linking and higher monomer elution. In our study, the lowest rate of reduction in weight percentage was seen in X-tra fil composite, when the tip of the light curing unit was in direct contact with the composite surface. It

indicates a better degree of polymer cross-linking in this group during polymerization and lower rate of monomer elution. On the other hand, X-tra base composite cured at 7 mm distance showed the highest percentage of weight loss at temperatures below 210°C. Similar to our study, some studies showed that the first drop in the weight loss curve in terms of temperature before reaching to composite degradation, was related to monomer evaporation, and by lowering this amount, the monomer content of the cured sample decreased, indicating a better degree of conversion of the composite during polymerization [19, 33].

Finally, the results of the TGA and GC in our study were congruent, which means that the higher the polymer cross-link density and thermal stability, the better the degree of polymerization and the lower the monomer elusion rate would be. In accordance with our results, a study conducted by Leprince et al. [31] showed that the filler weight percentage measured by the TGA was 85.2% and 74.2% in X-tra fil and X-tra base composites, respectively, and all physico-mechanical properties of X-tra fil composite were better than those of X-tra base composite. Furthermore, the degree of conversion using Raman spectrophotometric analysis was reported to be 62.1% and 58.6% for X-tra fil and X-tra base composites, respectively, confirming the results of our study.

CONCLUSION

Under the limitations of the present in vitro study, X-tra fil composite showed a significantly higher degree of conversion than X-tra base composite during the polymerization process, independent of the curing distance. Samples cured at 7 mm distance showed a significantly lower degree of conversion compared with those cured at 0 mm distance. The lowest monomer elution rate and higher degree of conversion were noted in X-tra fil composite cured at 0 mm distance.

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CONFLICT OF INTEREST STATEMENT

None declared.

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