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Original research

The effect of preheating on microhardness and flexural strength of bulk-fill resin composites: an *in-vitro* study

Mehdi Abed Kahnamouei[1]
Mahmoud Bahari[1,2]
Mohammad Esmaeel Ebrahimi Chaharom[1]
Soodabeh Kimyai[1]
Mehdi Daneshpooy[1]
Katayoun Katebi[3]
Bahareh Elhami[1]

Purpose
The objective of this study was to assess the impact of preheating on the microhardness and flexural strength of bulk-fill resin composites.

Materials and Methods
In this in vitro study, forty-two specimens were prepared of each composite, X-tra fil and Opus Bulk Fill, resulting in 84 disk-shaped specimens for microhardness testing and 84 bar-shaped specimens for flexural strength analysis. The specimens were divided into four groups as follows: Group 1: X-tra fil composite with preheating (at 68°C for 15 minutes), group 2: X-tra fil composite without preheating (at room temperature), group 3: Opus Bulk Fill composite with the same preheating method, group 4: Opus Bulk Fill composite without preheating. Microhardness was assessed using the Vickers test with a diamond indenter, and flexural strength was measured using a 3-point flexural test. Statistical comparisons were performed on the calculated results.

Results
In the preheated groups, both X-tra fil and Opus Bulk Fill composites exhibited significantly higher mean flexural strength compared to the non-preheated groups ($p<0.001$). However, there was no significant difference in the mean microhardness between the two groups for either type of composite ($p=0.719$). Additionally, the mean flexural strength and microhardness of X-tra fil composite, in both preheated and non-preheated conditions, were higher than those of the Opus Bulk Fill composite ($p<0.001$).

Conclusion
Preheating bulk-fill composites to 68°C has no detrimental effect on their microhardness and increases the flexural strength of these materials. Furthermore, the degree of microhardness and flexural strength in bulk-fill composites varies between brands and is influenced by their chemical compositions.

Keywords: *Composite resins, heating, x-tra fil composite resin, vickers test, microhardness*

Introduction

One of the recently introduced restorative materials is bulk-fill composite resins, in which the rate of polymerization shrinkage is reduced, and the depth of cure is increased up to 4 mm (1,2). Reduction in restoration time and an increase in the depth of cure have led to the widespread use of bulk-fill composites (3). Also, by increasing the bonding ability of bulk-fill composites to dentin, the marginal compatibility of the restoration has also improved (4). On the other hand, placing a block of these materials prevents voids formation and results in a dense restoration (5).

The high viscosity and stickiness of composite systems can cause problems during placement and adaptation (6). One of the proposed methods to solve this problem is preheating the composite, which leads

ORCID IDs of the authors: M.A.K. 0000-0002-0458-6366; M.B. 0000-0002-1774-7659; M.E.E.C. 0000-0002-8163-8510; S.K. 0000-0003-0703-5479; M.D. 0000-0002-1549-2488; K.K. 0000-0002-6595-6359; B.E. 0009-0009-3035-2967

[1]*Esthetic and Restorative Dentistry, Department of Esthetic and Restorative Dentistry, Faculty of Dentistry, Tabriz University of Medical Sciences, Tabriz, Iran*

[2]*Dental and Periodontal Research Center, Tabriz University of Medical Sciences, Tabriz, Iran*

[3]*Department of Oral and maxillofacial medicine, Faculty of Dentistry, Tabriz University of Medical Sciences, Tabriz, Iran*

Corresponding Author: Katayoun Katebi

E-mail: k_katebi@yahoo.co.uk

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to a reduction in viscosity, improved adaptation of the composite, reduced film thickness, and better handling (7,8). Composite preheating is recommended for all types of composite restorations, especially in deep cavities of posterior teeth where polymerization and adaptation in the deep layers of the material are of concern (9). Temperature has a significant effect on polymerization efficiency. Higher mobility of monomers due to increased temperature facilitates the connection between polymer chains and leads to improvement of mechanical and physical properties of composites, such as increased flexural strength and surface hardness (10).

Preheating of composites may be done by placing compoules or syringes of composite resin material in a composite heater or a water bath (11). Several studies have shown that preheating has no negative effect on the mechanical properties of nanohybrid and microhybrid resin composites (12,13). Also, preheating resin composites may increase polymerization, decrease shrinkage forces, and improve surface microhardness (14). Mechanical properties of restorative materials are of paramount importance as they directly impact their durability. Surface hardness, one of the key characteristics, is positively correlated with compressive strength, resistance to intraoral stresses, and the degree of conversion. When a material has low surface hardness, it is more susceptible to wear, which can result in restoration failure (15). Previous studies have demonstrated that preheating does not affect the flexural strength of bulk-fill and conventional composites. Additionally, the microhardness of bulk-fill composites remains unaffected by preheating. However, one study indicated that preheating does not impact the polymerization of bulk-fill composite resins, but it does enhance the microhardness of these composites (10,16,17).

Given the limited information available on bulk-fill composites and the influence of preheating on their properties, this in vitro study aims to investigate the effect of preheating on the microhardness and flexural strength of X-tra fil and Opus Bulk Fill composites. The null hypotheses tested in this study were that there are no differences in the microhardness and flexural strengths of the pre-heated and untreated composite samples.

Materials and Methods

Ethical approval

This project received Ethical approval with code (IR.TBZMED.VCR.REC.1400.316).

Sample size determination

To determine the sample size, the microhardness values were obtained from the study of Lucey *et al.* (18) and the flexural strength values were obtained from the study of Abdulmajeed *et al.* (16) Considering 95% confidence, 80% test power, two-tailed test, using G-Power version 3.1.9.6, the minimum sample size in each group was calculated as seventeen. To increase the study power, the sample size increased to 21 samples in each group (20% increase). And a total of 168 samples were used in this study.

Composite materials ans study groups

Two types of bulk fill composites, X-tra Fil (VOCO, Cuxhaven, Germany) Universal color and Opus Bulk Fill APS (FGM, Joinville-SC, Brazil) A1 color were selected for microhardness study. The characteristics of the composites are presented in Table 1. 84 microhardness disk-shaped samples were prepared based on the type of composite (X-tra fil or Opus Bulk Fill) and the preparation temperature (24°C or 68°C) in 4 groups (Figure 1), each group containing 21 samples, with a diameter of 4 mm and a thickness of 2 mm. Group 1: X-tra fil composite with preheating (the composites were placed for 15 minutes in a thermostatically controlled water bath set to 68 °C) Group 2: X-tra fil composite at room temperature Group 3: Opus Bulk Fill composite with preheating (similar to group 1) Group 4: Opus Bulk Fill composite without preheating (similar to group 2).

Polymerization

The composites were packed in an aluminum mold with 4 mm diameter and 2 mm thickness. A plastic strip and a glass slide were placed on them to remove the excess material. Then they were cured with a LED light cure device (Demetron A2, Kerr, Orange, CA, USA) at an intensity of 1000 mw/

Table 1: *Specifications of composite resin materials used in the study*

Material	X-tra fil	Opus Bulk Fill
Type	High viscosity bulk-fill	High viscosity bulk-fill
Color	Universal	A1
Filler size	2-3 micrometer	0.7-10 micrometer
Filler loading	86%w/70%v	76%w/58%v
Organic matrix	Bis-GMA, UDMA, TEGDMA	Urethanedimethacrylic monomers, Co- initiator, Stabilizers
Inorganic filler	*	Silanized silicon dioxide
Manufacturer	VOCO, Cuxhavan, Germany	FGM, Joinville, Brazil
Lot Number	2016174	240120

*The type of fillers has not been provided by the manufacturer.

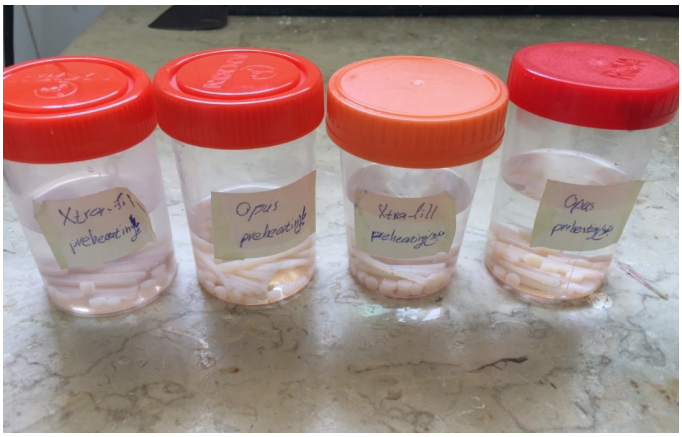

Figure 1. *Insertion The samples in 4 groups from left to right: X-tra fil composite with preheating, Opus Bulk Fill composite with preheating, X-tra fil composite without preheating, Opus Bulk Fill composite without preheating.*

cm2 for 40 seconds. After polymerization, the samples were separated from the mold and 600-grit silicon carbide abrasive was used to trim the excess material, and the dimensions of all samples were evaluated using a digital caliper (Super Caliper; Mitutoyo Corporation, Kanagawa, Japan).

Microhardness and flexural strength measurements

After fixing the samples in a holder, the surface was set perpendicular to the pyramidal intender with a square base. The surface microhardness of the samples was measured by Vickers test using a diamond intender (Innovatest, Micro Vickers tester, Micro-Met II, Buehler, IL, USA) with a load of 50 g for 15 seconds. An indented microscope with an eyepiece lens of 40 magnification was used to measure the indentations. Three depressions were created for each test sample and their mean was calculated (19).

To measure the flexural strength,84 rod-shaped samples were divided in four group based on the type of composite (X-tra fil or Opus Bulk Fill) and preparation temperature (24°C or 68°C), each group containing 21 square sectioned samples 25 mm long. They were prepared with dimensions of 2 x 2 mm. Flexural strength was determined by the ISO 4049 three-point flexural test. This test was performed using universal testing machine (H5K-S; Hounsfield Test Equipment, Redhill, UK) at a crosshead speed of 0.5 mm/min. The following formula was used to calculate the flexural strength: $\sigma = 3FL/2wt$; where F = maximum force applied; L = distance between the support beams; w = width of the specimen; and t = thickness of the specimen (16)

Statistical analysis

The results were reported by descriptive statistics mean and standard deviation. The Shapiro-Wilk test was used to test the distribution of quantitative variables. In order to compare microhardness and flexural strength between preheated and control groups, due to the normality of the dependent variable, analysis of covariance (ANCOVA) was used. A probability value of less than 0.05 was considered significant. Statistical Package for Social Sciences (SPSS) for Windows, version 16.0 (SPSS Inc., Chicago, IL, USA) was utilized for statistical analysis.

Results

The mean flexural strength in X-tra fil composite group was higher than the mean flexural strength in Opus Bulk Fill composite in both preheated and non-preheated modes ($p<0.001$) (Table 2). Also, in the preheated group the mean flexural strength was higher than the group without preheating ($p<0.001$) in both types of X-tra fil composite and Opus Bulk Fill. The interaction effect of Composite/Preheating was not significant in the mean flexural strength ($p=0.860$). Figure 2 shows the amount of flexural strength based on the type of composite and the intervention performed. The mean microhardness in X-tra-fil composite group in both modes was higher than the mean microhardness in Opus Bulk Fill composite group ($p<0.001$) (Table 3). There wasn't any significant difference in the microhardness of two groups with and without preheating ($p=0.719$). The interaction effect of Composite/ Preheating was not significant in the mean microhardness ($p=0.532$). Figure 3 shows the microhardness based on the type of composite and the intervention performed.

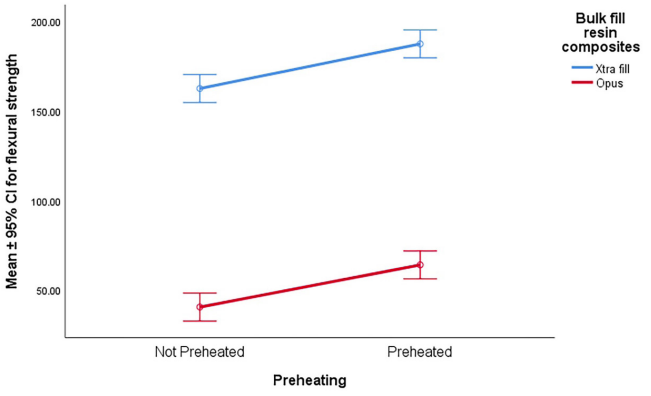

Figure 2. *Flexural strength based on the type of composite and the intervention.*

Table 2: *Flexural strength of X-tra fil and Opus Bulk Fill resin composites with and without preheating*

Composite	Preheating	Mean	SD	P-value		
				Composite	Preheating	Composite /Preheating
X-tra fil	Not Preheated	162.50	26.24	<0.001	<0.001	0.860
	Preheated	187.41	15.77			
Opus Bulk Fill	Not Preheated	40.59	11.70			
	Preheated	64.11	14.69			

SD: Standard Deviation

Table 3: *Microhardness of resin of X-tra fil and Opus Bulk Fill composites with and without preheating. SD: Standard Deviation)*

Composite	Preheating	Mean	SD	P-value		
				Composite	Preheating	Composite/ Preheating
X-tra fil	Not Preheated	73.63	6.34	<0.001	0.719	0.532
	Preheated	73.32	5.67			
Opus Bulk Fill	Not Preheated	35.08	4.17			
	Preheated	36.25	5.38			

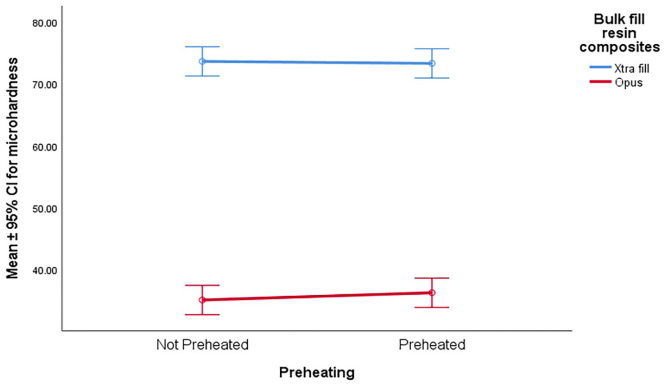

Figure 3. *Microhardness based on the type of composite and the intervention.*

Discussion

In the present study, preheating did not have any significant effect on the microhardness of X-tra fil and Opus Bulk Fill composites. In the study conducted by Degirmenci and Can (10), microhardness increased in Bulk Fill composites due to preheating, while microhardness in micro-hybrid composites decreased. Another study by Theobaldo *et al.* (17) found that preheating had no impact on the microhardness of Surefil SDR bulk composite. Interestingly, even though the conventional flow composite Filtek Z350 (used as a control group) had a higher volume percentage of filler (55%), its lower monomer-to-polymer conversion degree compared to the preheated bulk fill flow composite resulted in similar microhardness values. Therefore, it's evident that various factors influence the microhardness of composite resin materials.

In Lucey *et al.*'s study (18), preheating increased the microhardness of the hybrid (conventional) Spectrum TPH composite, which contrasts with the findings of the present study. This discrepancy can be attributed to the differences in the studied composites. Additionally, Nada and El-Mowafy (20) reported in their study that the effect of preheating on the surface microhardness of composites depends on the composite brand, its chemical composition, and particularly the type of monomer used. In the current study, preheating increased the flexural strength of both X-tra fil and Opus Bulk Fill composites. A study by Deb *et al.* (13) observed a significant increase in the flexural strength of the hybrid composite Spectrum TPH and Flow composite SDI after preheating, which was attributed to increased molecular activity in the polymer system and enhanced crosslinking in polymer chains. Kramer *et al.* (21) found that preheating increased the flexural strength of Filtek Supreme XT (conventional) nanocomposite and Tetric-Evo Cream bulk fill composite. The higher conversion degree of preheated composite resin was thought to have a positive effect on flexural strength. Alshali also reported that preheating composites before curing can increase polymerization and degree of conversion by temporarily reducing viscosity. In Abdulmajeed et al.'s study (16), preheating did not affect the flexural strength of Filtek One BulkFill composite and Filtek Supreme Ultra conventional composite, in contrast to the results of the present study. The discrepancy may be attributed to the differences in composite brands.

In the current study, both microhardness and flexural strength of X-tra fil bulk composite were higher than those of Opus Bulk Fill composite both before and after preheating. Alshali *et al.* (22) and Nag *et al.* (23) showed that microhardness values have a direct relationship with the amount of inorganic filler. They also demonstrated that the morphology and distribution of filler particles, particle shape and density, the ratio of monomer, type of monomer, crosslinking of polymers, and degree of conversion (DC) can account for variations in the microhardness of different resin composites. It was mentioned in Nag *et al.*'s study (23) that the manufacturers of X-tra fil bulk-filled composite increased the size of filler particles and filler content to enhance its microhardness. As a result, the microhardness of X-tra fil composite with 86% filler content was higher than that of Opus Bulk Fill composite with 79% filler content, both before and after preheating. Furthermore, in Degirmenci and Can's study (10), Estelite Bulk Fill Flow composite (EST), which contains BisGMA monomer, exhibited higher microhardness values than G-aenial Posterior and SDR Plus composites. BisGMA is a monomer with high molecular weight, strong hydrogen bonding capacity, and low molecular mobility. It is considered the most viscous and least flexible among dental resin monomers. The strong intermolecular bonding between hydroxyl groups in BisGMA likely contributed to the higher microhardness of EST, similar to the findings in the present study, where X-tra fil bulk composite containing BisGMA exhibited higher microhardness than Opus Bulk Fill composite.

Gomes *et al.* (24) demonstrated that the mechanical properties of bulk-filled composite resin depend on their filler content, with higher filler volume corresponding to higher flexural strength. In the current study, X-tra fil composite with 86% filler by volume had higher flexural strength than Opus Bulk Fill composite with 79% filler by volume. Additionally, the study by Deb *et al.* (13) identified an inverse relationship between flexural strength and shrinkage under preheating conditions. Therefore, composites with less shrinkage, such as X-tra fil, tend to exhibit higher flexural strength.

Microhardness in resin composites can be measured using either the Vickers or Knoop tests. Both methods involve creating an indentation with a diamond tip under a predefined force for a specified duration (25). In the present study, the Vickers test was employed for surface microhardness testing. Research has indicated a very strong correlation (r=0.991) between the Knoop and Vickers hardness tests (26). The Knoop test is based on linear measurements, while the Vickers test measures values based on an area. As a result, it is challenging to determine which test is more accurate (27), and either test can be used for material comparisons. The present study utilized X-tra fil and Opus Bulk Fill composites. These two brands were selected based on their relative acceptance and popularity among clinicians.

It is important to note that the present study examined only two brands of composite and two preheating temperatures, with samples analyzed 24 hours after curing. Future studies may explore other brands of bulk-fill composites, a wider range of preheating temperatures, and different post-curing timeframes, which could yield varying results. Additionally, the non-anatomical geometry of the samples, prepared according to ISO standards, may have influenced the results. To better simulate clinical conditions, the samples were polymerized from one side only. Moreover, this in vitro study was conducted without dental tissue, and results may differ in clinical settings. Future studies could investigate the effect of preheating on dental tissue and pulp by testing composites with dental tissue involved.

Conclusion

In light of the findings from this study, it can be stated that preheating bulk-fill composites to a temperature of 68 degrees Celsius does not compromise their microhardness. In fact, it results in an increase in their flexural strength. Additionally, substantial variations in both microhardness and flexural strength are observed among different bulk-fill composites, primarily stemming from differences in their chemical compositions.

Türkçe özet: *Ön ısıtmanın yığın dolgulu reçine kompozitlerin mikrosertliği ve eğilme mukavemeti üzerindeki etkisi: in vitro bir çalışma. Amaç: Bu çalışmanın amacı, ön ısıtmanın yığın dolgulu reçine kompozitlerin mikrosertliği ve eğilme mukavemeti üzerindeki etkisini değerlendirmektir. Gereç ve Yöntem: Bu in vitro çalışmada, X-tra fil ve Opus Bulk Fill kompozitlerinin her birinden kırk iki örnek hazırlandı; sonuçta mikro sertlik testi için 84 disk şeklinde örnek ve eğilme mukavemeti analizi için 84 çubuk şeklinde örnek elde edildi. Örnekler şu şekilde dört gruba ayrıldı: Grup 1: Ön ısıtmalı X-tra fil kompozit (68°C'de 15 dakika), grup 2: Ön ısıtmasız X-tra fil kompozit (oda sıcaklığında), grup 3: Opus Aynı ön ısıtma yöntemine sahip Bulk Fill kompoziti, grup 4: Ön ısıtmasız Opus Bulk Fill kompoziti. Mikro sertlik, elmas uçlu Vickers testi kullanılarak değerlendirildi ve bükülme mukavemeti, 3 noktalı bükülme testi kullanılarak ölçüldü. Hesaplanan sonuçlar üzerinde istatistiksel karşılaştırmalar yapıldı. Bulgular: Ön ısıtmalı gruplarda hem X-tra fil hem de Opus Bulk Fill kompozitleri, ön ısıtmasız gruplarla karşılaştırıldığında önemli ölçüde daha yüksek ortalama eğilme mukavemeti sergiledi (p<0.001). Ancak her iki kompozit türü için de iki grup arasında ortalama mikrosertlik açısından anlamlı bir fark yoktu (p=0,719). Ayrıca, X-tra fil kompozitinin ortalama eğilme mukavemeti ve mikrosertliği, hem ön ısıtmalı hem de ön ısıtmasız koşullarda, Opus Bulk Fill kompozitininkinden daha yüksekti (p<0.001). Sonuç: Bulk-fill kompozitlerin 68°C'ye kadar önceden ısıtılmasının mikrosertlikleri üzerinde zararlı bir etkisi yoktur ve bu malzemelerin eğilme mukavemetini arttırır. Ayrıca, toplu dolgulu kompozitlerdeki mikro sertlik ve bükülme mukavemetinin derecesi markalar arasında farklılık gösterir ve kimyasal bileşimlerinden etkilenir. Anahtar kelimeler: kompozit reçineler, ısıtma, x-tra fil kompozit reçine, vickers testi, mikrosertlik*

Ethics Committee Approval: This project has been reviewed and approved by the Ethics Committee of Tabriz University of Medical Sciences (IR.TBZMED.VCR.REC.1400.316).

Informed Consent: Not required.

Peer-review: Externally peer-reviewed.

Author contributions: MAK, MB, SK participated in designing the study. MB, MD, BE participated in generating the data for the study. MEEC, KK, BE participated in gathering the data for the study. MAK, MB, MEEC, MD participated in the analysis of the data. SK, BE wrote the majority of the original draft of the paper. MAK, MB, MEEC, SK, MD, KK, BE participated in writing the paper. MAK, MB, MEEC, SK, MD, KK, BE has had access to all of the raw data of the study. MAK, MB, KK has reviewed the pertinent raw data on which the results and conclusions of this study are based. MAK, MB, MEEC, SK, MD, KK, BE have approved the final version of this paper. MAK, KK guarantees that all individuals who meet the Journal's authorship criteria are included as authors of this paper.

Conflict of Interest: The authors declared that they have no conflict of interest.

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