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Research Article

EFFECT OF EXTENDED POLYMERIZATION TIMES ON THE DEGREE OF CONVERSION AND MICROHARDNESS OF RESIN COMPOSITES

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ABSTRACT

Objective: This study is of significant importance as it evaluates the polymerization properties of bulk-fill and conventional resin composites regarding the degree of conversion (DC) and microhardness (MH) at different polymerization times. The findings of this research can potentially influence the future of restorative dentistry.

Materials and Methods: In this study three different polymerization times (20 s, 60 s, and 100 s) were applied to disc-shaped samples (6 mm wide; and 2 mm high) prepared from two bulk-fill resin composites (Filtek One Bulk Fill Restorative, X-tra Fil) and two traditional resin composites (Filtek Z550, Charisma Smart). The DC of the polymerized samples was measured with a Fourier Transform Infrared/ Attenuated Total (FT-IR/ATR)device, and the MH values were measured with a Vickers hardness device. The collected data were subjected to statistical analysis.

Results: The FT-IR analysis and Vickers microhardness test results demonstrated that the DC and MH values of the groups exposed to 100 s of light curing were significantly higher than those of the other groups ($p<0.05$).

Conclusions: The extended polymerization time applied to the resin composites in this study significantly increased the materials' DC and MH.

Keywords: Bulk fill composite, Degree of conversion, Microhardness, Resin composite

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INTRODUCTION

At present, resin composites represent the most widely utilized restorative materials in dental practice for treating dental hard tissue defects (caused by caries, trauma, systemic disease, congenital factors, etc.) to restore the aesthetic and functional functions of teeth by ensuring the integrity and continuity of the dental tissue with correct diagnosis and appropriate treatment of the defects (1). The maximum thickness that can be applied to ensure adequate polymerization in traditional resin composites is 2 mm (2). For this reason, conventional resin composites are applied incrementally in deep cavities. In addition to requiring technical precision, incremental application is time consuming and there is a risk of contamination and gaps between layers (3). To overcome these limitations, manufacturers have developed bulk-fill resin composites that can be applied in 4-5 mm increments by incorporating modifications such as the use of alternative photoinitiators, adjustments in the size of inorganic fillers, and alterations in the monomer composition (4).

Adequate polymerization of resin composites is essential for the success of restorations (5). The physicomechanical properties of resin composites depend on the degree of polymerization; and, thus, on certain variables such as the color, content, and thickness of the composite, light polymerization unit, light intensity, wavelength and polymerization time (6, 7). Ideally, all the monomers in resin composites should be converted to polymers during the polymerization reaction. However, dimethacrylate monomers cannot polymerize completely and residual monomers (double carbon bonds) may remain, which can cause irreversible damage to the pulp through the dentinal tubules (8).

As the degree of polymerization increases, the amount of residual monomer that does not participate in the reaction in the organic matrix decreases, which enhances the physical properties of the material, such as the elastic modulus, color stability, biocompatibility, solubility and monomer release (9, 10). As the DC increases and the material's residual monomer content decreases, the resin composite's hardness increases (11). An indicator of the high DC of resin composites is that they reach sufficient hardness values . When the literature is scanned, microhardness tests are frequently used as an indirect method when determining DC (12).

While previous studies have compared the microhardness and monomer conversion rates of bulk-fill and conventional resin composites, the effect of extended light exposure time has yet to be thoroughly investigated (13, 14). This study aimed to assess the impact of extended polymerization times on the DC and MH of two different bulk-fill and two conventional resin composites. The null hypothesis of our study is as follows:

1. The DC does not change as the light application time increases.
2. MH does not change as the light application time increases.

MATERIALS AND METHODS

In this study, one traditional nanohybrid resin composite (FZ550; Filtek™ Z550, 3M ESPE, St Paul, MN, USA), one traditional microhybrid resin composite (CS; Charisma Smart, Heraeus Kulzer GmbH, Hanau, Germany) and two bulk fill resin composites (FOBF; Filtek™ One Bulk Fill Restorative, 3M ESPE, St Paul, MN, USA) and (XF; X-tra Fil, Voco, Cuxhaven, Germany) were used.

A power analysis was conducted to determine the appropriate sample size. According to the power analysis results of the test performed in the G*Power 3.1 program with an alpha value of 0.05 and an 85% confidence interval, the minimum number of samples was 5. For this reason, the number of samples in our study was determined to be 6.

Specimen preparation

In our study, resin composites were applied to a Teflon mold (6 mm in diameter, 2 mm in height) placed on glass with the help of a handpiece. Then, another glass was placed under constant hand pressure to provide a flat form to the upper surface and prevent air bubbles from remaining. To achieve equal light distance standards for each resin composite sample, the tip of the light device was positioned in direct contact with the glass and perpendicularly. The prepared samples were polymerized with an LED light device (Valo / Cordless, Ultradent Products Inc, South Jordan, UT, USA) for 20 s (control group), 60 s and 100 s. After the samples were prepared, finishing and polishing procedures were performed in a dry condition the Sof-Lex Composite Finishing and Polishing Disc Set (3M ESPE, St Paul, MN, USA) sequentially from coarse to fine grit with a low-speed handpiece. Each disc was used for 30 seconds and every two samples were changed. The prepared samples were categorized according to their groups, placed in dry conditions within lightproof containers, and stored at room temperature for 24 hours. The DC of the composite samples was evaluated via FT-IR/ATR analysis, and the

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MH values were evaluated via Vickers hardness measurements.

Vickers microhardness

The samples (12 groups n=6) were placed under the Vicker notch tip of the microhardness device (EMCO Test/DuroScan, Kellau, Kuchl, Austria). A load of 100 g (2,942 N) was applied to the samples for 15 s, and three measurements of each sample were made at three different points. The mean of the three measurements was calculated and recorded as the hardness value of each sample. Rectangular-shaped notches were made with a Vicker's notching tip positioned perpendicular to the surfaces of the samples. After the notch was opened, the diagonal lengths of the quadrangular notches formed in the samples placed under the x40 magnification lens of the microhardness device were manually determined with the help of the arms moving in the x-y-z plane of the hardness device, and the device automatically calculated the Vickers hardness value.

FT-IR/ATR analysis

The spectra of the polymerized samples (12 groups, n=6) for which the DC of the monomer was measurements were measured with an FT-IR/ATR device (Perkin Elmer/ 400 FT-IR/ATR Spectrometer Spotlight 400 Imaging System, Waltham, Massachusetts, USA) at 4000-400. In the cm^{-1} wavenumber range, 20 scan counts and 4 cm^{-1} wavenumber resolution were recorded. First, FT-IR/ATR spectra of non-polymerized composite samples were recorded. Subsequently, the polymerized composite samples were positioned against the ATR crystal, and the device's clamping arm was secured. In this way, spectral measurements of each sample were performed, and the absorbance values were measured. The DC was calculated by substituting the obtained data and the determined absorbance values of the double-bonded carbons into the formula below.

$$DC\ (\%) = \left(1 - \frac{\left(\frac{\text{aliphatic}}{\text{aromatic}}\right)\text{polymer}}{\left(\frac{\text{aliphatic}}{\text{aromatic}}\right)\text{monomer}}\right) * 100$$

Statistical analysis

While the findings obtained in the study, were being evaluated the SPSS 20 (SPSS Inc.; Chicago, IL, USA) program was used for statistical analysis. Statistical analyses of the data obtained from the samples studied with FT-IR/ATR and Vicker's Hardness devices were performed via one-way variance (One-Way ANOVA) to evaluate the differences between groups, and two-way variance (Two-Way ANOVA) to assess the effects of various times and materials was performed. Multiple comparisons were performed using Bonferroni and Tukey HSD post-hoc analyses. A p-value of less than 0.05 was considered statistically significant for all analyses.

RESULTS

The mean values and standard deviations for the DC and MH of the resin composites evaluated in this study are presented in Table 1. Based on the calculated results, extending the polymerization time significantly enhanced the DC and MH values of the resin composites tested ($p < 0.05$). The MH values of the FOBF and CS resin composites increased as the polymerization time increased, but the difference was not statistically significant ($p>0.05$). When the MH values of individual resin composites were examined during the polymerization period, FZ550 presented the highest value, and the CS resin composite presented the lowest value.

Table 2 displays the resin composites' DC and MH values and standard deviations, regardless of the light curing duration. When we looked at the DCs of the resin composites used regardless of the polymerization time, XF had the highest value, and the CS resin composite had the lowest value.

Table 1. Average degree of conversion (DC) and microhardness (MH) findings and standard deviations of resin composites according to light application time

			Degree of Conversion		Microhardness	
Resin Composite	**Time**	**N**	**Mean DC±SD**	**Statistical Difference**	**Mean MH±SD**	**Statistical Difference**
	20 s	6	50.7±0.7	A	74.6±8.3	A
FZ550	60 s	6	55.8±1.3	B	109.5±13.7	B, C
	100 s	6	68±1.5	C	112.4±14.4	B, C
	20 s	6	27.2±0.8	D	97.1±14.8	B
CS	60 s	6	35±1.5	E	103.5±16.8	B
	100 s	6	39.8±1.4	F	126.1±4.4	C
	20 s	6	30.4±1.1	G	59.1±13	A, D
FOBF	60 s	6	32.2±0.8	G	59.5±20.4	A, D
	100 s	6	49.6±1.1	A	75.6±16	A
	20 s	6	59.8±0.8	H	39.8±6.6	D
XF	60 s	6	65.2±0.5	I	54.3±15.4	A, D
	100 s	6	65.6±0.8	I	58.2±8.6	A, D

The DC and MH values and standard deviations of the resin composites used with different polymerization times regardless of the type of composite are shown in Table 3.

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Table 2. Average degree of conversion (DC) findings and standard deviations of the resin composites used regardless of the light application time

Resin Composite	N	Degree of Conversion Mean DC±SD	Degree of Conversion Statistical Difference	Microhardness Mean MH±SD	Microhardness Statistical Difference
FZ550	18	57.9±8.8	A	108.9±15.2	A
CS	18	34±6.3	B	50.8±9.6	B
FOBF	18	37.4±10.6	C	64.7±9.4	C
XF	18	63.5±3.2	D	98.8±21	D

Table 3. Average degree of conversion (DC) findings and standard deviations of resin composites due to light application, regardless of the resin composite type

Time	N	Degree of Conversion Mean DC±SD	Degree of Conversion Statistical Difference	Microhardness Mean MH±SD	Microhardness Statistical Difference
20 s	24	42± 15.07	A	67.65± 24.2	A
60 s	24	47.1± 16.1	B	81.7± 28.8	B
100 s	24	55.6± 13.3	C	93± 31.5	C

DISCUSSION

In our study, increasing the polymerization time of bulk-fill and traditional resin composites from 20 s to 100 s significantly increased their DC. There was no statistically significant difference between the 20 and 60 s polymerization times of the FOBF samples and the 60 and 100 s polymerization times of the XF samples. Therefore, according to our findings, the first null hypothesis of the study, which was that the DC would not changes as the light application time increased, was rejected.

It is hypothesized that the DC of resin composites is directly correlated with the duration of light-curing exposure (15). In our study, the impact of varying light curing durations on the DC across different resin composite materials was investigated, and it was observed that increasing the light application time from 20 seconds to 100 seconds resulted in increased DC in the samples. Lempel et al. (13) and Szalewski et al. (15) compared the DC of resin composite samples by polymerizing them for different periods; and a positive correlation existed between the duration of light curing and the DC. These data in the literature support the results of our study.

The literatüre has reported that DC is affected by the type of monomers in the organic matrix and their viscosity (16). Yıldırım et al. (17), and Szczesio-Wlodarczvk et al. (18) in their study comparing the DC of methacrylate-based resins, reported that the highest DC in TEG-DMA and the lowest DC in Bis-GMA. They also found the DC order was Bis-GMA < Bis-EMA < UDMA < TEGDMA. Manufacturers combine Bis-GMA with other monomers to increase the DC of resin composites by reducing viscosity. In particular, the presence of amine groups in the UDMA monomer increases the mobility of radical sites through characteristic chain transfer reactions that provide a second pathway for the continuation of polymerization. Thus, DC has increased (16).

Although both the FZ550 and CS groups used in our study are traditional resin composites, the reason different DCs may be due to the differences in the compositions of the materials. The manufacturer reported that the CS composite contains only Bis-GMA in its matrix. Its low DC can be explained by the high amount of this high-viscosity compound in its organic matrix. FZ550 contains UDMA, Bis-EMA and TEG-DMA in addition to Bis-GMA. Since these monomers decreased the viscosity of Bis-GMA and increased monomer mobility, they may have caused an increase in DC.

In our study, the FOBF groups generally presented low DC in all periods. A study evaluating the DC of different types of resin composites polymerized by applying light for a standard time, suggested that FOBF had the lowest DC, possibly due to its organic matrix content (14). FOBF contains different monomers such as high molecular weight AUDMA, AFM, and DDDMA, which can increase the stiffness of the polymer chain. High molecular weight monomers reduce the number of reactive groups on the organic matrix and inhibit their mobility during the polymerization reaction. This information supports the results of the monomer conversion degree obtained by looking at the monomer content of the resin composites we used in the study.

Since bulk-fill resin composites are applied at relatively high thicknesses, their opacity is reduced and their translucency is increased by reducing the amount of inorganic fillers in their structure for increased light transmission and sufficient polymerization (19). It is claimed in the literature that a high filler ratio negatively affects DC. It is thought that increasing the number of inorganic fillers causes the interaction of monomers, especially those with high viscosity, such as Bis-GMA, to weaken. As a result, it becomes difficult for the material to polymerize (5, 21). In contrast, in our study, the amount of inorganic fillers in the resin composites used was between 76% and 86%. According to our results, the percentage of DC in FZ550 (81.8%) was greater than that of the other samples. These results showed that the amount of inorganic fillers alone may not have much effect on DC, and may be due to the geometry of the inorganic fillers or differences in their components.

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Microhardness testing is another commonly employed method to assess the DC in resin composites. In our study, as the duration of light application increased, it increased in MHs as in DCs. Therefore, our second null hypothesis was also rejected. MH is affected by the inorganic filler content of resin composites. Adding zirconium, barium and ytterbium particles to the inorganic filler structures of resin composites to increase their radiopacity also increases their light transmittance (22). Tekçe et al. (23) suggested that the high inorganic filler ratio and the presence of Zr/SiO_2 particles effectively increase the MH value of the FZ550 resin composite. In line with the data we obtained in parallel with this information, the significant superiority in the MS values of XF and FZ550 can be attributed to the high Zr/SiO_2 ratio in the inorganic filler.

Nagi et al. (24) compared the MH values of two bulk-fill resin composites polymerized at different thicknesses (2, 3 and 4 mm) and for various times (10, 20, 40 and 60 s). The XF group had higher hardness values than the other resin composites. It has been suggested that the XF bulk-fill resin composite shows high MH due to the large size and amount of inorganic filler particles. However, no statistically significant differences were detected in the MH values of the XF bulk fill resin composite across varying light curing durations. In our study, XF groups with a thickness of 2 mm to which light was applied for different periods, presented high MH values. In contrast, there was a statistically significant difference in the MH values at 20 s compared with those at 60 s and 100 s.

This study's limitations include its in vitro design, which does not account fort he effects of temperature, humidity, and oral fluids. Additionally, the optical properties of dental tissues, such as their ability to reflect and transmit light were not considered. The impact of the type of light source used was not evaluated, and the effects of different light sources should be further investigated. Moreover, the study examined only specific polymerization durations, which constitutes another significant limitation..

CONCLUSION

Considering the limitations of the present study, it can be concluded that the resin composite XF exhibited the highest degree of conversion (DC) regardless of light exposure duration, while FZ550 showed the highest DC specifically at 100 seconds of light application. Furthermore, Vickers microhardness (MH) testing revealed that FZ550 had the highest MH values among the tested composites. Overall, both the degree of conversion and microhardness generally increased with extended light exposure up to 100 seconds.

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Authorship contributions
Concept: SD, HB, Design: SD, HB Data collection or processing: TE Analysis and interpretation: HB, TE Literature search: TE Writing: HB, TE.

Data availibity statement
The data supporting this study's findings are available from the corresponding author upon reasonable request.

Declaration of competing interest
There is no conflict of interest in this study.

Ethics
This study does not require ethical committee approval.

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REFERENCES

1. Fahad F, Majeed MA. Fracture resistance of weakened premolars restored with sonically-activated composite, bulk-filled and incrementally-filled composites (A comparative in vitro study). J Bagh Coll Dent 2014; 26, 22-27.
2. Alqudaihi FS, Cook NB, Diefenderfer KE, Bottino MC, Platt JA. Copmarision of internal adaptation of bulk-fill and increment-fill resin composite materials. Oper Dent 2019; 44(1): E32-E44.
3. Karadas M, Hatipoğlu O, Er H, Akyüz Turumtay E. Influence of different light-curing units on monomer elution from bulk fill composites. JAST 2018; 32(23): 2631-2646.
4. Yu P, Yap A, Wang X. Degree of conversion and polymerization shrinkage of bulk-fill resin-based composites. Oper Dent 2017; 42(1), 82-89.
5. Obeid AT, Kojic DD, Felix C, Velo MM, Furuse AY, Bombonatti JF. Effects of radiant exposure and distance on resin-based composite polymerization. Am. J. Dent 2022; 35(4): 172-177.
6. Algamaiah H, Silikas N, Watts DC. Conversion kinetics of rapid photo-polymerized resin composites. Dent Mater J 2020; 36(10): 1266-1274.

7. Gonulol N, Ozer S, Tunc ES. Effect of a third - generation LED LCU on microhardness of tooth - colored restorative materials. Int J Paediatr Dent 2016; 26(5), 376-382.
8. Odabaşı D, Guler C, Kucukaslan D. Evaluation the amount of residual monomer released from different flowable composite resins. BMC Oral Health 2024; 24(1): 244.
9. Sarosi C, Moldovan M, Soanca A, Roman A, Gherman T, Trifoi et al. Effects of monomer composition of urethane methacrylate based resin on the C=C degree of conversion residual monomer content and mechanical properties. Polymers 2021; 13(24):
10. Moldovan M, Balazsi R, Soanca A, Roman A, Sarosi C, Prodan D et al. Evaluation of the degree of conversion, residual monomers and mechanical properties of some light-cured dental resin composites. Materials 2019; 12(13):
11. Hatipoğlu Ö, Par M, and Hatipoğlu FP. Comparison of degree of conversion performance of bulk-fill resin composites: A systematic review and network meta-analysis of in vitro studies. J Dent 2024; 105289.
12. El-Maksoud OA, Hamama HHH, Wafaie RA, El-Wassefy N, Mahmoud SH. Effect of shelf-storage temperature on degree of conversion and microhardness of composite restorative materials. BMC Oral Health 2023; 23(1):57.
13. Lempel E, Öri Z, Szalma J, Lovász BV, Kiss A, Tóth Á et al. Effect of exposure time and pre-heating on the conversion degree of conventional, bulk-fill, fiber reinforced and polyacid-modified resin composites. Dent Mater 2019; 35(2): 217-228.
14. Strini BS, Marques JFDL, Pereira R, Sobral-Souza DF, Pecorari VGA, Liporoni PCS et al. Comparative evaluation of bulk-fill composite resins; knoop microhardness, diametral tensile strength and degree of conversion. Clin Cosmet Investig Dent 2022; 225-233.
15. Szalewski L, Wójcik D, Sofińska-Chmiel W, Kuśmierz M, Różyło-Kalinowska I. How the duration and mode of photopolymerization affect the mechanical properties of a dental composite resin. Materials 2022; 16(1): 113
16. Deng H, Liu F, He J. The Effect of Inorganic Filler Content on the Properties of BPA-Free Bulk-Fill Dental Resin Composites. Materials 2024; 17(20): 5040.
17. Yıldırım ZS, Eyiler E, Kürklü ZGB. Effect of thickness on the degree of conversion, monomer elution, depth of cure and cytotoxicity of bulk-fill composites. J. Oral Sci 2023; 65(2): 121-126.
18. Szczesio-Wlodarczyk A, Domarecka M, Kopacz K, Sokolowski J, Bociong K. An evaluation of the properties of urethane dimethacrylate-based dental resins. Materials 2021; 14(11): 2727.
19. Sampaio CS, Abreu JLBD, Kornfeld B, Silva EMD, Giannini M, Hirata R. Short curing time bulk fill composite systems: volumetric shrinkage, degree of conversion and Vickers hardness. Braz. Oral Res 2024; 38: e030.
20. Haugen HJ, Marovic D, Par M, Khai Le Thieu M, Reseland JE, Johnsen GF. Bulk fill composites have similar performance to conventional dental composites. Int. J. Mol. Sci 2020; 21(14): 5136.
21. Duman AN, Cevik P, Doğan A. The Effect of the Resin Type and Filling Placement Techniques on the Degree of Conversion of Various Resin-Based Composites. Appl. Sci 2024; 14(23): 11215.
22. Pedreira PR, Damasceno JE, Cerqueira GAD, Souza AF, Aguiar FHB, Marchi GM. Radiopacity and physical properties evaluation of infiltrants with Barium and Ytterbium addition. Braz. Dent. J. 2023; 34: 93-106.
23. Tekçe N, PALA K, Özel E, Tuncer S, Demirci M. Üç Farklı Kompozit Materyalinin Yüzey Sertliği Üzerinde Polimerizasyon Süresinin Etkisi. Turkiye Klinikleri Journal of Dental Sciences 2015; 21(2).
24. Nagi SM, Moharam LM, Zaazou MH. Effect of resin thickness, and curing time on the micro-hardness of bulk-fill resin composites. J Clin Exp Dent 2015; 7(5), e600.