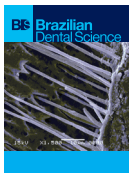
Brazilian Dental Science
Ciência Odontológica Brasileira
unesp
UNIVERSIDADE ESTADUAL PAULISTA "JÚLIO DE MESQUITA FILHO"
Instituto de Ciência e Tecnologia
Campus de São José dos Campos



ORIGINAL ARTICLE

Doi: 10.14295/bds.2020.v23i2.1915

Effect of storage time and composite thickness on Degree of Conversion of Bulk-fill and universal composites using FTIR method

Efeito do tempo de armazenamento e da espessura no grau de conversão de compósitos Bulk-fill e universais usando o método FTIR

Farnaz FARAHAT[1], Abdolrahim DAVARI[2], Marzieh FADAKARFARD[3]

1 - Department of Operative Dentistry - School of Dentistry - Shahid Sadoughi University of Medical Sciences - Yazd - Iran.

2 - Department of Operative Dentistry - Member of Social Determinant of Oral Health Research Center - School of Dentistry - Shahid Sadoughi University of Medical Sciences - Yazd - Iran.

3 - Shahid Sadoughi University of Medical Sciences - Yazd – Iran.

ABSTRACT

Objective: Degree of Conversion (DC) of Resin-Based composites (RBCs) is a concern and is affected by different factors. This study was conducted to evaluate the effect of different thicknesses and storage times on DC of bulk-fill and universal RBCs using the FTIR method. **Material and Methods:** For each of dental composite (Tetric N- Ceram Bulk Fill and Tetric N Ceram), 28 samples were prepared (14 samples with 2 mm height and 14 samples with 4 mm height). One and seven days (1d and 7d) after storage in an incubator at 37 ˚C, DC was measured and recorded using a FTIR device. Data were analyzed using t-test and Three-Way ANOVA and Tukey Post-Hoc Test. $P < 0.05$ was considered statistically significant. **Results:** DC of Bulk-fill composite is greater than universal composite. DC at 2 mm thickness is more than 4 mm. Unlike universal composite, 7d DC of Bulk-fill composite is greater than 1d. Of course, none of these differences are statistically significant (P-value > 0.05). **Conclusion:** Considering the limitation of this study, since no difference was found between DC of 1d and 7d, 1d DC is sufficient for clinical procedures, such as polishing, and the laboratory tests.

KEYWORDS

Bulk-fill composites; Degree of Conversion; FTIR.

RESUMO

Objetivo: O grau de conversão (DC) de compósitos à base de resina (RBCs) é um assunto que merece cuidado e é afetado por diferentes fatores. Este estudo foi conduzido para avaliar o efeito de diferentes espessuras e tempos de armazenamento no DC de compósitos Bulk-fill e compósitos universais usando o método FTIR. **Material e Métodos:** Para cada compósito dentário (Tetric N-Ceram Bulk Fill e Tetric N Ceram), foram preparadas 28 amostras (14 amostras com 2 mm de altura e 14 amostras com 4 mm de altura). Um e sete dias (1d e 7d) após o armazenamento em uma incubadora a 37 ° C, o DC foi mensurado e registrado usando um dispositivo FTIR. Os dados foram analisados por meio do teste t, ANOVA três fatores e teste Post-Hoc de Tukey. $P < 0,05$ foi considerado estatisticamente significante. **Resultados:** O DC do compósito Bulk-fill é maior que o compósito universal. DC com 2 mm de espessura é superior a 4 mm. Ao contrário do compósito universal, o DC com 7d do Bulk-fill é maior que 1d. Obviamente, nenhuma dessas diferenças é estatisticamente significante (valor P> 0,05). **Conclusão:** Considerando a limitação deste estudo, uma vez que não foi encontrada diferença entre as DC de 1d e 7d, a 1d DC é suficiente para procedimentos clínicos, como polimento e testes laboratoriais.

PALAVRAS-CHAVE

Compósitos Bulk-fill; Grau de conversão; FTIR

INTRODUCTION

Recently, regarding the advance of Resin Based Composite (RBC) and bonding methods, and the increasing tendency to prepare the conservative cavities, RBCs are widely used to restore posterior teeth [1]. For many years, the use of layering technique with a thickness of up to 2 mm has been considered as a standard [2]. But this technique is time-consuming and creating bubbles or contamination between different layers of composite is likely. Recently, Bulk-Fill (BF) RBCs have been marketed. According to the manufacturers' claims, these composites can be inserted in deep cavities in a single layer of 4-5 mm without the need for a longer curing time or more intense light exposure. This advantage will result in shorter restoration time [3].

The Degree of Conversion (DC) is an important factor for the proper functioning of RBCs. It was found that there is a direct correlation between DC and the physical and mechanical properties of RBCs [4,5]. The reported DC of BF is different. Ilie [6] reported DC of two types of BF as 41.4 % and 43.8 %. Another study showed DC 43.6 % to 76.5 % for nine BF RBCs [7]. Curing depth and DC of BF are measured using several methods such as Micro-hardness, Fourier Transform Infrared Spectroscopy (FTIR) and Raman Spectroscopy. Some studies have found that DC of BF is sufficient in 4 mm thickness [2,8-10], But some investigations reported that all BF RBCs were not sufficiently cured in the 4-mm bulk [11,12].

Although most RBC polymerization occurs in the first 20 s, post-cure polymerization continues slowly after light curing. Generally, it is thought that post-cure polymerization is completed within 24 hours after light curing [9,13]. Post-cure polymerization of RBCs after 24 hours is rarely investigated. A study has reported the gradual increase in composite hardness during 7 days [9] and another research has shown an increase in composite DC after 30 days [14]. Only one study examined post-cure polymerization of BF and showed that their DC increased during 7 days [15].

Therefore, the purpose of this study is to evaluate the effect of different thicknesses (2 mm and 4 mm) and storage times (1d and 7d) on DC of bulk-fill and universal RBCs using FTIR method. The study null hypothesis includes:

1) the RBC type has no effect on DC;

2) the composite thickness does not affect DC;

3) no difference in DC is found between two different storage times (1 d and 7 d).

MATERIAL AND METHODS

The materials studied in the present study are two nano-hybrid RBCs with A2 shade: 1) Tetric N Ceram Bulk-Fill (TNCBF), 2) Tetric N Ceram (TNC). The full specifications of them are given in table I.

Table I - Materials used in this study

Material	Shade	Type of filler	Filler (weight %)	Filler (vol%)	Resin matrix	Manufacture	Batch Nomber
Tetric N-Ceram	A2	Barium glass, Ytterbium, Triflouride,Mixed oxid and copolymer	80-81	55-57	Dimethacrylate	Ivoclar Vivadent-Italy	T24404
Tetric N-Ceram Bulk fill	A2	Barium Glass, Ytterbium, Trifluoride, Prepolymer and Mixed Oxide	75-77	53-55	Dimethacrylate	Ivoclar Vivadent-Italy	T29061

In order to prepare the samples, Teflon molds (5 mm diameter× 2 mm or 4 mm height) were used. A thin Mylar Strip (Kerr Hawe Neosdent, Bioggio, Switzerland) and a mold were put on a glass slab. The composites were packed bulky into the mold to reach the desired height. Next, a Mylar strip and another glass slab were placed on the mold, and a gentle finger pressure was applied to remove excess materials. After that the glass slab was

removed from the mold; samples were light cured for 20 s from the top using Light-Emitting Diode (LED) (Demi Plus, Kerr Hawe Neosdent, Bioggio, Switzerland) with head diameter of 8 mm and intensity 800 mW/cm^2 on continuous mode. For each RBCs, 14 samples with 2 mm height and 14 samples with 4 mm height were prepared. In each thickness, half of the samples were stored 1 day in an incubator (Behdad, Tehran, Iran) in water at 37 ˚C, and other samples were stored 7 days. To evaluate DC of samples, they were powdered and were analyzed with a FTIR spectrometer (IR Prestige-21, Shimadzu, Duisburg, Germany). The absorbance spectrum was acquired by scanning the specimens 10 times over a 1670–1550 cm-1 range with a resolution of 4 cm^{-1}. The DC was then calculated by comparing the height of the peaks for the methacrylate vinyl group in the cured material against that in the uncured material, using the following formula:

$$DC\ \%=1-\frac{[Abs\ (aliphatic)/\ Abs\ (aromatic)]\ polymer}{[Abs\ (aliphatic)/\ Abs\ (aromatic)]\ monomer}$$

In which the peak height around 1638 cm-1 and 1608 cm-1 indicating the absorbance intensities of aliphatic and aromatic C=C, respectively.

The collected data entered software SPSS-22 (SPSS Inc., IL, USA) and analyzed by Three-way ANOVA, T-test and Tukey Post-Hoc test. The statistical significance was considered as 0.05.

RESULTS

The mean values of DC in RBC samples are shown in Table II. According to Table II, the highest mean value of DC belongs to Group 6 and the lowest value belongs to Group 4. The mean DC of BF and universal RBCs was compared using T-test. The results showed that the mean DC of TNCBF is greater than TNC. But this difference is not statistically significant (P-value = 0.425).

Table II - Mean and standard deviation(SD) of DC in studied groups

Composite	thickness (mm)	Storage time (day)	Group number	number	Mean ± SD (%)
Tetric N-Ceram	2	1	1	7	53.07 ± 8.25
		7	2	7	45.25 ± 8.17
	4	1	3	7	47.77 ± 26.36
		7	4	7	43.44 ± 13.18
Tetric N-Ceram Bulk fill	2	1	5	7	48.25 ± 12.63
		7	6	7	55.09 ± 5.00
	4	1	7	7	48.92 ± 4.68
		7	8	7	50.39 ± 16.65

In the study of the effect of thickness on DC by t-test it was found that DC of 2 mm thickness is greater than 4 mm, but this difference was not statistically significant (P-value=0.498). Storage time had also no significant effect on DC (P-value = 0.815).

Three-way ANOVA and Tukey Post-Hoc were used to study the simultaneous effect of two variables on the mean DC of RBCs. unlike TNC, 7d DC of TNCBF is more than 1d.

DISCUSSION

The present study has examined DC of conventional and BF RBCs with different thicknesses and storage times. According to the study results, the mean DC of BF is more than universal, the mean DC of BF and universal RBCs with 2 mm thickness is more than 4 mm. Also, DC of BF at 7d was more than 1d, but in universal the result was reversed. Of course, none of these differences were significant. Therefore, all of our three null hypotheses were confirmed.

Different methods are used for measuring DC, among which FTIR is the most appropriate and reliable method widely used [16]. FTIR measures C=C bond modulation before and after the light curing that is the most sensitive method for evaluating DC in RBCs [17].

Regarding the possibility of increasing DC of composite restoration up to 24 hours after the light curing [18-20] and the significant effect of temperature on DC of

composites [21], in the present study, in order to obtain more accurate results, all DCs were measured after 24 hours storing of cured samples in a dark environment at 37 ˚C. Also, in the present study, light curing device was located tangential on the upper part of the mold, which is usually impossible in clinical conditions. Therefore, DC values in clinical conditions may be less than lab study [22,23].

In the present study, DC of TNCBF was 48.25-55.09% and DC of TNC was 43.44-53.07 % (Table II). The normal DC for a clinically acceptable restoration has not been determined [17], however, various studies have considered DC 35-80 % as desirable [24]. Therefore, all DCs obtained in the present study are acceptable. It seems that the difference in the results of various studies can be attributed to several factors such as various time intervals between sample preparation and laboratory test, type of the mold, the curing methods as well as the temperature conditions and the storage environment of the samples after preparation [25].

According to the study results, DC of BF is greater than universal. This result is consistent with many of the previous studies [3,26-28]. Of course, the results of this study, unlike other studies, are not statistically significant. P Yu et al. concluded that with increasing depth, BF shows better DC than other RBCs. In their study, DC after 24 hours with 2 mm thickness for TNC and TNCBF was 59.78 % and 56.22 %, respectively and 56.11 % and 55.46 % with 4 mm thickness, respectively, which are close to each other like the present study as well as no significant difference was found between two thicknesses of two RBC types [29].

Several factors such as size and composition of the filler, translucency, light intensity, curing time, monomer composition and initiator concentration can have a significant effect on DC of RBCs [30]. In filler structure of TNCBF, pre-polymerized particles (PPF) containing inorganic barium glass and silica are used, which have a good effect on radiation energy absorption and polymerization. Due to application of PPF, RBCs like TNCBF are able to achieve a high filler load while maintaining a low specific surface between inorganic fillers and organic matrix [3,23,27,28].

The lack of coordination between refractive index of filler and matrix lead to light scattering and reducing absorbed optical energy by RBC and consequently the restoration DC. Hence, one of the most suitable methods for increasing the composite restoration DC is to increase its translucency by establishing the coordination between the refractive index of filler and matrix [20]. According to the claims of the manufacturers, TNCBF has higher translucency than TNC, which allows for more (about 15 %) light penetration [29].

The type of photo-initiator of RBCs will have a significant effect on their DC [31,32]. In recent years, studies indicate the low ability of camphorquinone (CQ) initiator to create free radicals and lower sensitivity to light than some of newer RBCs [25,33]. For example, Ivocerin initiator added to TNCBF, based on Moszner [22] and Bucuta [27] studies, is more effective than CQ. This phenomenon is due to the fact that in Ivocerin, germanium is used which is more sensitive to light than other initiators. Therefore, the use of Ivocerin can be attributed to higher DC of BF than universal.

According to the results of the present study, the mean DC of two RBCs with 2 mm thickness is more than 4 mm. Of course, this difference is statistically insignificant. The RBCs are polymerized to a depth that light is penetrated, so with increasing thickness, light cannot penetrate into the depth of the composite. A study showed that all BF RBCs could have a high DC at up to 3 mm thickness [34]. In a study by Jain et al., in all BF RBCs, DC decreased from 2 mm to 4 mm thickness [17]. However, another study using FTIR to determine the DC at 1mm depths of

two types of BF RBCs showed that in Smart Dentin Replacement (SDR) composite, DC of the samples remained unchanged in 1-4 mm thicknesses [15]. However, a study showed that by increasing composite thickness, more curing time is needed to obtain the desired DC [10]. Various studies have shown that DC level reduces from top surface to bottom surface [17]. However, in the present study, DC of powdered sample was measured under FTIR, which could represent the actual DC of each 2 mm or 4 mm layer.

In the present study, no significant difference was found between two storage times. In most studies, it has been shown that 5 min after light curing, composite DC may not be adequate and some post polymerization conversion can occur within 24 hours [29]. With increasing the viscosity of the composite mass, the polymerization reaction rate is reduced and so post-cure polymerization continues slowly until free radicals become immobilized. Therefore, DC values do not reach 100 % [15].

A study by Par et al. showed that some of the chemical components used in some of BF RBCs may increase DC after 7 days. They stated that their study results should not be generalized to all types of BF RBCs, because each RBC has different components [15]. According to the results of our study, since no difference is found between DC of 1d and 7d, 1d DC is sufficient for clinical procedures, such as polishing, and the laboratory study of mechanical properties of BF RBCs. However, further studies examining other BF RBCs and effect of different shades, initiators and fillers on DC are needed.

CONCLUSION

Regarding the limitations of the present study and determination of DC using FTIR, the following can be deduced:

- No significant difference is found between DC of TNBF and TNC;
- In both RBC, after 7d, DC did not have a significant difference with 1d, so procedures such as polishing and laboratory tests can be done 1d after light curing;
- Composite thickness and storage time have not a significant effect on DC of bulk-fill and universal RBCs.

REFERENCES

1. Sadowsky SJ. An overview of treatment considerations for esthetic restorations: a review of the literature. J Prosthet Dent. 2006;96(6):433-42. doi: 10.1016/j.prosdent. 2006.09.018.
2. Tarle Z, Attin T, Marovic D, Andermatt L, Ristic M, Tauböck TT. Influence of irradiation time on subsurface degree of conversion and microhardness of high-viscosity bulk-fill resin composites. Clin Oral Investig. 2015:19(4):831-40. doi: 10.1007/s00784-014-1302-6.
3. Ilie N, Bucuta S, Draenert M. Bulk-fill resin-based composites: an in vitro assessment of their mechanical performance. Oper Dent. 2013;38(6):618-25. doi: 10.2341/12-395-L.
4. Alshali RZ, Silikas N, Satterthwaite JD. Degree of conversion of bulk-fill compared to conventional resin-composites at two time intervals. Dent Mater. 2013;29(9):e213-7. doi: 10.1016/j.dental.2013.05.011.
5. Lohbauer U, Rahiotis C, Krämer N, Petschelt A, Eliades G. The effect of different light-curing units on fatigue behavior and degree of conversion of a resin composite. Dent Mater. 2005;21(7):608-15. doi: 10.1016/j.dental.2004.07.020.
6. Ilie N, Keßler A, Durner J. Influence of various irradiation processes on the mechanical properties and polymerisation kinetics of bulk-fill resin based composites. J Dent. 2013;41(8):695-702. doi: 10.1016/j.jdent.2013.05.008.
7. Leprince JG, Palin WM, Vanacker J, Sabbagh J, Devaux J, Leloup G. Physico-mechanical characteristics of commercially available bulk-fill composites. J Dent. 2014;42(8):993-1000. doi: 10.1016/j.jdent.2014.05.009.
8. Rizzante FAP, Duque JA, Duarte MAH, Mondelli RFL, Mendonça G, Ishikiriama SK. Polymerization shrinkage, microhardness and depth of cure of bulk fill resin composites. Dent Mater J. 2019;38(3):403-410. doi: 10.4012/dmj.2018-063.
9. Alrahlah A, Silikas N, Watts D. Post-cure depth of cure of bulk fill dental resin-composites. Dent Mater. 2014;30(2):149-54. doi: 10.1016/j.dental.2013.10.011.
10. Farahat F, Daneshkazemi A, Hajiahmadi Z. The effect of bulk depth and irradiation time on the surface hardness and degree of cure of bulk-fill composites. J Dent Biomater. 2016;3(3):284-91.
11. Jang J, Park S, Hwang I. Polymerization shrinkage and depth of cure of bulk-fill resin composites and highly filled flowable resin. Oper Dent. 2015;40(2):172-80. doi: 10.2341/13-307-L.
12. Garcia D, Yaman P, Dennison J, Neiva G. Polymerization shrinkage and depth of cure of bulk fill flowable composite resins. Oper Dent. 2014;39(4):441-8. doi: 10.2341/12-484-L.
13. Al-Ahdal K, Ilie N, Silikas N, Watts DC. Polymerization kinetics and impact of post polymerization on the Degree of Conversion of bulk-fill resin-composite at clinically relevant depth. Dent Mater. 2015;31(10):1207-13. doi: 10.1016/j.dental.2015.07.004.
14. Leung R, Fan P, Johnston W. Post-irradiation polymerization of visible light-activated composite resin. J Dent Res.1983;62(3):363-5.
15. Par M, Lapas-Barisic M, Gamulin O, Panduric V, Spanovic N, Tarle Z. Long term degree of conversion of two bulk-fill composites. Acta Stomatol Croat. 2016;50(4):292-300. doi: 10.15644/asc50/4/2.

16. Moraes LG, Rocha RS, Menegazzo LM, de Araújo EB, Yukimito K, Moraes JC. Infrared spectroscopy: a tool for determination of the degree of conversion in dental composites. J Appl Oral Sci. 2008 Mar-Apr;16(2):145-9. DOI: 10.1590/s1678-77572008000200012

17. Jain L, Mehta D, Meena N, Gupta R. Influence of light energy density, composite type, composite thickness, and postcuring phase on degree of conversion of bulk-fill composites. Contemp Clin Dent. 2018;9(Suppl 1):S147- S152. doi: 10.4103/ccd.ccd_169_18.

18. Truffier-Boutry D, Demoustier-Champagne S, Devaux J, Biebuyck J-J, Mestdagh M, Larbanois P, et al. A physico-chemical explanation of the post-polymerization shrinkage in dental resins. Dent Mater. 2006;22(5):405-12. doi: 10.1016/j.dental.2005.04.030.

19. Stansbury JW. Dimethacrylate network formation and polymer property evolution as determined by the selection of monomers and curing conditions. Dent Mater. 2012;28(1):13-22. doi: 10.1016/j.dental.2011.09.005.

20. Skrtic D, Antonucci JM. Effect of chemical structure and composition of the resin phase on vinyl conversion of amorphous calcium phosphate-filled composites. Polym Int. 2007;56(4):497-505. doi: 10.1002/pi.2129.

21. Price RB, Whalen J, Price TB, Felix CM, Fahey J. The effect of specimen temperature on the polymerization of a resin-composite. Dent Mater. 2011;27(10):983-9. doi: 10.1016/j.dental.2011.06.004.

22. Moszner N, Fischer UK, Ganster B, Liska R, Rheinberger V. Benzoyl germanium derivatives as novel visible light photoinitiators for dental materials. Dent Mater. 2008;24(7):901-7. doi: 10.1016/j.dental.2007.11.004.

23. Arikawa H, Kanie T, Fujii K, Takahashi H, Ban S. Effect of filler properties in composite resins on light transmittance characteristics and color. Dent Mater J. 2007;26(1):38-44. doi: 10.4012/dmj.26.38.

24. Sakaguchi RL, Powers JM. Craig's restorative dental materials.13th ed. Philadelphia: Elsevier; 2012. 172p.

25. Bhamra G, Fleming G, Darvell B. Influence of LED irradiance on flexural properties and Vickers hardness of resin-based composite materials. Dent Mater. 2010;26(2):148-55. doi: 10.1016/j.dental.2009.09.008.

26. Flury S, Hayoz S, Peutzfeldt A, Hüsler J, Lussi A. Depth of cure of resin composites: is the ISO 4049 method suitable for bulk fill materials? Dent Mater. 2012;28(5):521-8. doi: 10.1016/j.dental.2012.02.002.

27. Bucuta S, Ilie N. Light transmittance and micro-mechanical properties of bulk fill vs. conventional resin based composites. Clin Oral Investig. 2014;18(8):1991-2000. doi: 10.1007/s00784-013-1177-y.

28. Czasch P, Ilie N. In vitro comparison of mechanical properties and degree of cure of bulk fill composites. Clin Oral Investig. 2013;17(1):227-35. doi: 10.1007/s00784-012-0702-8.

29. Yu P, Yap A, Wang X. Degree of conversion and polymerization shrinkage of bulk-fill resin-based composites. Oper Dent. 2017;42(1):82-9. doi: 10.2341/16-027-L.

30. Johnston WM, Leung R, Fan P. A mathematical model for post-irradiation hardening of photoactivated composite resins. Dent Mater. 1985;1(5):191-4.

31. Neshchadin D, Rosspeintner A, Griesser M, Lang B, Mosquera-Vazquez S, Vauthey E, et al. Acylgermanes: photoinitiators and sources for Ge-centered radicals. Insights into their reactivity. J Am Chem Soc. 2013;135(46):17314-21. doi: 10.1021/ja404433u.

32. Rueggeberg FA. State-of-the-art: dental photocuring--a review. Dent Mater. 2011;27(1):39-52. doi: 10.1016/j.dental.2010.10.021.

33. Leloup G, Holvoet P, Bebelman S, Devaux J. Raman scattering determination of the depth of cure of light-activated composites: influence of different clinically relevant parameters. J Oral Rehabil. 2002;29(6):510-5. doi: 10.1046/j.1365-2842.2002.00889.x

34. Gonçalves F, Campos LMdP, Rodrigues-Júnior EC, Costa FV, Marques PA, Francci CE, et al. A comparative study of bulk-fill composites: degree of conversion, post-gel shrinkage and cytotoxicity. Braz Oral Res. 2018 Mar 8;32:e17. doi: 10.1590/1807-3107bor-2018.vol32.0017

Marzieh Fadakarfard
(Corresponding address)
dahe fajr Blvd,Yazd, Iran
E-mail: marziefadakarfard1373@gmail.com

Date submitted: 2019 Oct 18
Accept submission: 2020 Jan 07