Effect of Light Curing Time and Depth of Cure on Degree of Polymerization of Bulk-Fill Resin Composites

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Abstract

Objectives: To investigate the effect of curing time on the degree of polymerization to bulk-fill resin composites, and to investigate the effect of depth of cure on the degree of polymerization of bulk-fill resin composites. *Methodology*: There were three types of bulk-fill resin composites used in this study which were Filtek^{TM} Bulk Fill, Tetric® N-Ceram and SureFil® SDR flow+. Each type of resin composites was prepared in 2, 4 and 5 mm. Then they were light-cured for 20, 40, and 60 seconds which could be divided into 27 groups with different types of bulk-fill resin composites, depths of cure and lengths of curing time. Each specimen was cured with light having intensity of 1,200 mW/cm^2 in conventional mode. All of the specimens were kept in artificial saliva where they were incubated at 37°C for 24 hours. After the incubation, all of the specimens were tested using Vickers microhardness tester on the top and bottom surfaces. Then the bottom-to-top-surface microhardness ratio were used to analyze the degree of polymerization of each bulk-Fill resin composites. The data were analyzed with One-way ANOVA and T-test. *Results and Discussion*: All types of bulk-fill resin composites in the study could be significantly improved in degree of polymerization when increased curing time from 20 to 40 seconds and from 40 to 60 seconds. As the depth of cure increased, it became more statistically significantly decreased in degree of polymerization. Therefore, the more curing time and less depth of cure, there was an improved degree of polymerization. *Conclusion*: This study found a positive relationship between the level of bottom-to-top-surface microhardness ratio and curing time. Moreover, there existed a strong negative relationship between bottom-to-top-surface microhardness ratio and depth of cure. Last but not least, this study found an interaction effect of curing time and depth of cure.

Keywords: *Bulk-fill resin composites, Degree of polymerization, Microhardness ratio*

1. Introduction

Recently with increasing demand for esthetic, tooth colored restorations like resin-based composites are used for posterior teeth restoration. Also, patient awareness toward the use of mercury restoration has increased demand for resin-based composites even more. Resin composites are the type of materials which has the broadest application in restorative dentistry due to its optical and physical properties being very similar to natural dental tissue. The material presents as its principal characteristic adhesion to the dental structure, avoiding further damage to dental tissue (Barros, Lins, & Martins, 2017). However, the conventional resin composites require the maximum thickness of 2 mm depth of cure, which should be placed with incremental techniques to achieve the appropriate polymerization in a deep cavity. The incremental technique requires more clinical time and might produce voids due to the placing of multiple increments. In order to save the procedure time, less pronounced void with low-polymerization contraction, there is an innovation created with a single placement called bulk-fill resin composites.

Microhardness measurement is an indirect method to evaluate the degree of the polymerization of resin composites (Lenug, Fan, & Jonston, 1983; Silva, & Dias, 2009). The level of hardness is referred to strength and resistance against a compressive force. The surface hardness of the resin composites relates to its resistance to deformation and the capability to remain stable. Many studies demonstrated significant positive correlation between hardness and degree of conversion (Ferracane, 1985; Lombardini, Chiesa, Scribante, Colombo, &

Poggio, 2012). In this study, we used the microhardness test to assess the degree of the polymerization of resin composites by using Vickers hardness tester. This method has been successfully used for measurement of microhardness by many previous studies (Ferracane, 1985; Lombardini, Chiesa, Scribante, Colombo, & Poggio, 2012; Ansari, Ghasemi1, Vatandoust, & Motamedi, 2016). The bottom to top surface microhardness ratio can be used as a simple method to assess for polymerization adequacy at a specific depth, which is independent to resin composites composition. Ratios ranging from 80% to 90% are implied as having adequate conversion at the specific specimen depth (Ferracane, 1985).

It is widely known that both curing time and depth of cure play an important role in affecting polymerization. According to the recommendation from 3M ESPE, Dentsply and Ivoclar Vivadent which are the three manufacturers producing bulk-fill resin composites used in this study, 20 second curing time is required at 4 mm depth. Moreover, all of these 3 manufacturers recommend that light intensity be greater than 1000 mW/cm^2 during curing process to achieve adequate polymerization (3M ESPE, 2015) (Dentsply Sirona, 2016) (Ivoclar Vivadent). It is important to choose the most optimal curing time and depth of cure so that patients would benefit from the best degree of polymerization of resin composites.

2. Objectives

1. To investigate the effect of curing time on the degree of polymerization of bulk-fill resin composites
2. To investigate the effect of depth of cure on the degree of polymerization of bulk-fill resin composites

3. Materials and Methodology

The following materials were used to conduct the research.

1. Cylindrical stainless-steel mold with 4 mm diameter which allows us to adjust the depth of cure to 2, 4 and 5 mm
2. Light curing unit (Bluephase®N) with the light intensity of 1,200 mW/cm^2
3. Mylar strip
4. Glass slab
5. Filtek™ Bulk Fill Restoratives Shade A3
6. Tetric® N-Ceram Bulk Fill Shade IVA
7. SureFil® SDR flow+ Shade A3
8. Vickers microhardness tester
9. Silicone jig

Composites were placed in a 4 mm diameters cylindrical stainless-steel mold in order to block the light source from the external environment. The mold was adjustable in different depths of cure which were 2, 4 and 5 mm. The specimens were prepared from 3 types of bulk-fill resin composites which were conventional bulk-fill resin composites (Filtek™ Bulk Fill), conventional bulk-fill resin composites (Tetric® N-Ceram Bulk Fill) and flowable bulk-fill resin composites (SureFil® SDR flow+). Each type of bulk-fill resin composites was prepared in 2, 4 and 5 mm depths of cure. Then they were light-cured for 20, 40, and 60 seconds. Therefore, the specimens in the study could be divided into 27 groups with different types of bulk-fill resin composites, depths of cure and lengths of curing time.

After placing composites into the mold, the top surface of each specimen was covered by a piece of mylar strip. The tip of light curing unit was placed on each piece of mylar strip on each specimen. We fixed the position of each specimen which was adjacent to the tip of the light curing unit throughout the study.

Each specimen was cured with light having intensity of 1,200 mW/cm^2 in conventional mode. Then, all of the specimens were kept in artificial saliva where they were incubated at 37°C for 24 hours. After the incubation, we measured microhardness of each specimen using the Vickers microhardness test. For each specimen, we defined 5 testing points on the top surface and 5 testing points on the bottom surface. Then, we used the average level of microhardness that we had measured from 5 testing points to calculate the bottom-to-top-surface microhardness ratio.

In short, the microhardness ratio is the average level of microhardness on the bottom surface divided by the average level of microhardness on the top surface. It was used to measure the degree of polymerization of each specimen. We used One-way ANOVA and T-test to check whether the degree of polymerization was affected by curing time and depth of cure which are the independent variables in our study.

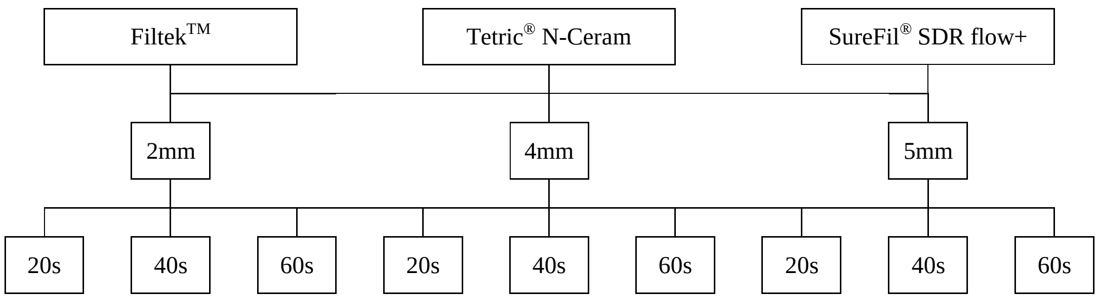

Figure 1 The diagram shows how specimens were divided.

4. Results and Discussion

Table 1 Means and standard deviations of the bottom-to-top-surface microhardness ratio of each type of bulk-fill resin composites at different periods of curing time and depths of cure. The numbers below are in percentage points

Type of resin composites	Curing time	Depth of cure		
		2 mm	4 mm	5 mm
Filtek™	20s	74.54 ± 1.97 $_{A,a}$	51.11 ± 1.36 $_{A,b}$	30.19 ± 0.79 $_{A,c}$
	40s	95.59 ± 1.02 $_{B,a}$	75.62 ± 1.95 $_{B,b}$	54.59 ± 1.79 $_{B,c}$
	60s	96.24 ± 0.75 $_{B,a}$	82.19 ± 1.42 $_{C,b}$	74.78 ± 1.49 $_{C,c}$
Tetric ® N-Ceram	20s	84.15 ± 1.01 $_{A,a}$	78.39 ± 2.85 $_{A,b}$	23.18 ± 1.49 $_{A,c}$
	40s	87.45 ± 1.82 $_{B,a}$	83.27 ± 1.48 $_{B,b}$	42.46 ± 1.69 $_{B,c}$
	60s	80.48 ± 0.85 $_{C,a}$	76.56 ± 0.36 $_{C,b}$	54.40 ± 1.10 $_{C,c}$
SureFil ® SDR flow+	20s	91.71 ± 1.65 $_{A,a}$	56.94 ± 3.51 $_{A,b}$	27.86 ± 1.13 $_{A,c}$
	40s	93.64 ± 2.56 $_{A,a}$	73.85 ± 1.45 $_{B,b}$	48.34 ± 1.33 $_{B,c}$
	60s	89.45 ± 1.49 $_{B,a}$	74.94 ± 1.74 $_{B,b}$	69.34 ± 2.26 $_{C,c}$

The difference in capital letters in the above table indicates statistical significance of difference in bottom-to-top-surface microhardness ratio at a fixed depth of cure, and varying periods of curing time (p-value ≤ 0.05). On the other hand, the difference in the lowercase letters represents statistical significance of the difference in bottom-to-top-surface microhardness ratio at a fixed period of curing time, and varying depths of cure (p-value ≤ 0.05).

Table 2 Means and standard deviations of the level of microhardness measured on the bottom surface of each type of bulk-fill resin composites at different periods of curing time and depths of cure. The numbers below are in percentage points

Type of resin composites	**Curing time**	**Depth of cure**		
		2 mm	4 mm	5 mm
Filtek™	20s	$43.67 \pm 0.77_{A,a}$	$29.92 \pm 1.01_{A,b}$	$17.21 \pm 0.54_{A,c}$
	40s	$55.12 \pm 2.03_{B,a}$	$45.68 \pm 0.98_{B,b}$	$32.77 \pm 1.22_{B,c}$
	60s	$57.40 \pm 1.19_{B,a}$	$49.96 \pm 0.65_{C,b}$	$45.02 \pm 1.04_{C,c}$
Tetric® N-Ceram	20s	$51.28 \pm 0.92_{A,a}$	$46.64 \pm 1.49_{A,b}$	$13.89 \pm 1.01_{A,c}$
	40s	$54.40 \pm 1.17_{B,a}$	$51.52 \pm 0.63_{B,b}$	$26.82 \pm 0.88_{B,c}$
	60s	$56.19 \pm 0.92_{C,a}$	$50.03 \pm 0.47_{B,b}$	$37.58 \pm 0.57_{C,c}$
SureFil® SDR flow+	20s	$30.32 \pm 0.91_{A,a}$	$17.37 \pm 0.78_{A,b}$	$8.58 \pm 0.38_{A,c}$
	40s	$30.74 \pm 1.03_{A,a}$	$23.30 \pm 0.46_{B,b}$	$16.09 \pm 0.58_{B,c}$
	60s	$31.75 \pm 0.47_{B,a}$	$24.39 \pm 0.37_{C,b}$	$23.20 \pm 0.94_{C,c}$

The difference in capital letters in the above table indicates statistical significance of difference in level of microhardness measured on the bottom surface of each specimen at a fixed depth of cure, and varying periods of curing time (p-value $\leq$0.05). On the other hand, the difference in lowercase letters represents statistical significance of difference in the level of microhardness measured on the bottom surface~~s~~ of each specimen at a fixed period of curing time, and varying depths of cure (p-value $\leq$0.05).

First of all, we could observe a positive relationship between the level of bottom-to-top-surface microhardness ratio and curing time even though some of the improvement of the degree of polymerization as curing time increased was not statistically significant. In other words, more curing time generally improved the degree of polymerization. Nonetheless, we observed 3 exceptional cases where the bottom-to-top-surface microhardness ratio decreased while we increased the curing time. Specifically, the bottom-to-top-surface microhardness ratio of Tetric® N-Ceram significantly decreased when we increased the curing time from 40 to 60 seconds at 2 and 4 mm depths of cure. Also, the bottom-to-top-surface microhardness ratio of SureFil® SDR flow+ significantly decreased when we increased the curing time from 40 to 60 seconds at 2 mm depth of cure. In spite of these 3 exceptional results, we could still conclude that in general more curing time improved the degree of polymerization. The reason was because the reduction of the bottom-to-top-surface microhardness ratio in these 3 exceptional cases was due merely to the rate of increase of the average level of microhardness on the bottom surface was less than the rate of increase of the average level of microhardness on the top surface as curing time increased. Recall that the microhardness ratio is the average level of microhardness on the bottom surface divided by the average level of microhardness on the top surface. When the rate of increase in the numerator is less than the rate of increase in the denominator, the value of the whole ratio drops. According to Table 2, the level of microhardness measured on the bottom surface of Tetric® N-Ceram did not significantly decrease when we increased curing time from 40 to 60 seconds at 2 and 4 mm depths of cure. In fact, the level of microhardness measured on the bottom surface of Tetric® N-Ceram actually increased when we increased the curing time from 40 to 60 seconds at 2 mm depth of cure. Also, the level of microhardness measured on the bottom surface of SureFil® SDR flow+ significantly increased when we increased curing time from 40 to 60 seconds at 2 mm depth of cure. Hence, we concluded that there existed a positive relationship between the level of bottom-to-top-surface microhardness ratio and curing time in our study.

Apart from the positive relationship between the level of bottom-to-top-surface microhardness ratio and curing time in our study, we could also observe a strong negative relationship between the bottom-to-top-surface microhardness ratio and depth of ~~the~~ cure. In other words, less depth of cure generally improved the quality of polymerization.

Furthermore, it is worth to point out that we could observe an interaction effect of curing time and depth of cure. Even though we used One-way ANOVA and T-test to check whether the quality of polymerization was affected by curing time and depth of cure and these 2 independent variables were treated independently, evidence of the interaction effect of curing time and depth of cure could be briefly seen in Table 1 and 2. It was noticeable from these tables that as depth of cure increased, we could see more statistically significant improvement of the degree of the polymerization as we increased curing time. To be more specific, for 2 mm depth of cure, Tetric ® N-Ceram was the only type of bulk-fill resin composites that we could observe constant statistically significant improvement of the degree of polymerization when we increased curing time from 20 to 40 seconds and from 40 to 60 seconds. For 4 mm depth of cure, Filtek™ and SureFil ® SDR flow+ were the two types of bulk-fill resin composites that we could observe constant statistically significant improvement of the degree of polymerization when we increased curing time from 20 to 40 seconds and from 40 to 60 seconds. For 5 mm depth of cure, all types of bulk-fill resin composites in the study could be significantly improved in degree of polymerization when we increased curing time from 20 to 40 seconds and from 40 to 60 seconds. Therefore, it could be inferred from the result there existed a higher degree of effect of curing time on the bottom-to-top-surface microhardness ratio as depth of cure increased.

The findings in our study coincide with many previous studies where they reported positive relationships between level of bottom-to-top-surface microhardness ratio and curing time (David, Gomes, Gomes, & Loguercio, 2007; Alpöz et al., 2008; Mousavinasab, & Meyers, 2009). However, there are some preceding studies where they reported that there existed no relationship between level of bottom-to-top-surface microhardness ratio and curing time (Okte, Villalta, Garcia-Godoy, Garcia-Godoy, & Murray, 2005; Flury, Hayoz, Peutzfeldt, Hüsler, & Lussi, 2012; Catelan et al., 2014). This is contradicting to the findings in our study.

Recall that ratios ranging from 80% to 90% are implied as having adequate conversion at the specific specimen depth of cure (Ferracane, 1985). Also, according to the recommendation from 3M ESPE, Dentsply and Ivoclar Vivadent which are the three manufacturers producing bulk-fill resin composites used in this study, 20 second curing time is required at 4 mm depth of cure. However, the results in our study show that 20 second curing time at 4 mm depth of cure was not enough to achieve the acceptable range of the ratio. The results in our study coincide with many previous studies where they also reported that some bulk-fill resin composites had adequate degree of polymerization when depths of cure were only up to 2 or 3 mm (Yap, Pandya, Toh, 2016; Gracia, Yaman, Dennison, Neiva, 2014).

5. Conclusion

We investigated a relationship between the degree of polymerization and curing time. We also investigated the relationship between the degree of polymerization and depth of cure. This study found a positive relationship between level of bottom-to-top-surface microhardness ratio and curing time. Moreover, there existed a strong negative relationship between bottom-to-top-surface microhardness ratio and depth of cure. Last but not least, this study found an interaction effect of curing time and depth of cure.

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7. References

Barros, R., Lins, E., & Martins, L. (2017). Bulk-Fill Resin-Based Composites. *Adv Dent & Oral Health*, *4*(5), 1-2.

Lenug, L., Fan, P., & Jonston, W. (1983). Post-irradiation polymerization of visible light- activated composite resins. *J Dent Res, 62*(3), 363-365.

Silva, CM., & Dias, KR. (2009). Compressive strength of esthetic restorative materials polymerized with quartz-tangsten- halogen light and blue LED. *Braz Dent J, 20*(1), 54-57.

Ferracane, JL. (1985). Correlation between hardness and degree of conversion during the setting reaction of unfilled dental restorative resins. *Dent Mater, 1*, 11–14.

Lombardini, M., Chiesa, M., Scribante, A., Colombo, M., & Poggio, C. (2012). Influence of polymerization time and depth of cure of resin composites determined by Vickers hardness. Dent *Res J, 9*(6), 735–740.

Ansari, Z., Ghasemi1, A., Vatandoust, M., & Motamedi, M. (2016). Effect of curing time on polymerization rate of bulk-fill composite resins. *Journal of Dental School*, 34(4), 214-224.

FiltekTM BulkFill Posterior restorative technical product profile. (2015). *FiltekTM Posterior Restorative 2015*. Retrieved from http://multimedia.3m.com/mws/media/976634O/filtek-bulk-fill-posterior-restorative-technical-product-profile.pdf

SDR flow+ Direction for Use & Technique Guide. (2016). *Dentsply Sirona Class ll total practice solution 2016*. Retrieved from https://www.class2restorations.com/dental-restorative-materials/bulk-fill-flowable/product-resources#product-resources

Tetric N-ceram instructions for use. *Ivoclar Vivadent*. Retrieved from http://downloads.ivoclarvivadent.com/zoolu-website/media/document/16422/Tetric+N-Ceram+Bulk+Fill?__hstc=22451044.0b94ac98b786b8334b0b73db75471d7c.1553386495886.1553386495886.1553386495886.1&__hssc=22451044.1.1553386495887&__hsfp=3515352052

David, JR., Gomes, OM., Gomes, JC., & Loguercio, AD. (2007). Reis A. Effect of exposure time on curing efficiency of polymerizing units equipped with light-emitting diodes. *J Oral Sci*, 49(1), 19-24.

Alpöz, AR., Ertugrul, F., Cogulu, D., Ak, AT., Tanoglu, M., & Kaya, E. (2008). Effects of light curing method and exposure time on mechanical properties of resin based dental materials. *Eur J Dent*, 2(1), 37-42.

Mousavinasab, SM., & Meyers, I. (2019). Curing efficacy of light emitting diodes of dental curing units. *J Dent Res Dent Clin Dent Prospects*, 3(1), 11-16.

Okte, Z., Villalta, P., Garcia-Godoy, F., Garcia-Godoy, F Jr., & Murray, P. (2005). Effect of curing time and light curing systems on the surface hardness of compomers. *Oper Dent*, 30(4), 540-545.

Flury, S., Hayoz, S., Peutzfeldt, A., Hüsler, J., & Lussi, A. (2012). Depth of cure of resin composites: is the ISO 4049 method suitable for bulk fill materials? *Dent Mater*, 28(5), 521-528.

Catelan, A., Pollard, T., Bedran-Russo, A., Santos, P dos., Aihorosano, G., & Aguiar, F. (2014). Light-curing time and aging effects on the nanomechanical properties of methacrylate – and silorane-based restorations. *Oper Dent*, 39(4), 389-397.

Yap, AU, Pandya, M, Toh, WS. (2016). Depth of cure of contemporary bulk‐fill resin‐based composites. *Dent Mater J*, 35(3), 503–510.

Garcia, D, Yaman, P, Dennison, J, Neiva, G. (2014). Polymerization shrinkage and depth of cure of bulk fill flowable composite resins. *Oper Dent*, 39(4), 441–448.