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RESEARCH ARTICLE

Comparison of Surface Microhardness of the Flowable Bulk-Fill Resin and the Packable Bulk-Fill Resin according to Light Curing Time and Distance

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Background: As a restorative material used to treat dental caries, the light-curing type resin is widely used, but it has the disadvantage of polymerization shrinkage. The Bulk-Fill composite resin was developed to solve these shortcomings, but the existing research mainly focused on comparing the physical properties of a composite resin and a Bulk-Fill resin. A study on the light curing time and distance of the Bulk-Fill resin itself tend to be lacking.

Methods: This study compares the surface microhardness of specimens prepared by varying the light curing time and distance of smart dentin replacement (SDR) as a flowable Bulk-Fill resin and Tetric N-ceram as a packable Bulk-Fill resin, and confirms the polymerization time and distance that becomes the optimum hardness. To determine the hardness of the specimen, it was measured using the Vickers Hardness Number (Matsuzawa MMT-X, Japan).

Results: In SDR, the surface microhardness decreased as the distance increased in all time groups in the change distance from the curing tip. In the change of light curing time with respect to the distance from curing tip, the surface microhardness increased as the time increased. In Tetric N-ceram, the surface microharness showed no significant difference in the change of the distance of curing tip in the group of 20 and 60 second. But in the group of 10 and 40 seconds, decreased as the distance increased. The surface microharness increased as the light curing time increased in all distance groups.

Conclusion: When using SDR and Tetric N-ceram in clinical practice, it is considered that as the distance from the polymerization reactor tip increases, a longer light curing time than the polymerization time recommended by the manufacturer is required.

Key Words: Flowable bulk-fill resin, Light curing distance, Light curing time, Packable bulk-fill resin, Surface microhardness

Introduction

1. Background

As a method of treating dental caries, a method of removing the caries and filling it with a tooth substitute material is used. There are many types of materials, but one of them is resin. In the past, light-curing resins were used only for restoration of small lesions or when aesthetics were required, but recently, not only aesthetics but also physical properties have been greatly improved, so they are widely used as replacements for extensive tooth loss[1]. A clinically successful condition for dental restoration is to ensure good adhesion to the tooth hard tissue. In the case of composite resins, the major drawback is polymerization shrinkage[2]. Postoperative hypersensitivity due to stress caused by polymerization shrinkage, microleakage and discoloration of the restoration, it causes various clinical problems such as tooth deformation[3]. In addition, there are disadvantages such as long working time, sensitivity to moisture and contamination, as well as polymerization shrinkage, which are more prominent when treating children[4]. In order to prevent polymerization

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and shrinkage of the composite resin, the layered filling method was used, but when layered filling is performed, there are problems in that the treatment time is prolonged even if the operator is highly skilled, and it is not easy to control moisture and contamination[5].

In order to solve these disadvantages, bulk filling appeared, and Bulk-Fill composite resins that made this possible were developed[4]. These Bulk-Fill composite resins have low shrinkage stress and shrinkage rate[6], and conventional resins recommend resin restorations up to 2 mm in height, but Bulk-Fill resins can restore up to 4～5 mm in height[7] and can be used as a single restorative material. It also has usability[8] and good marginal fit[9]. Bulk-Fill resins can be divided into flowable type and packable type (conventional type) based on viscosity according to filler content[3]. Van Ende et al.[10] found that the flowable type needs to cover the existing composite resin when used due to the low abrasion resistance of the surface, but the full-body Bulk-Fill (packable type) does not require additional filling of the existing composite resin unlike the flowable type.

These Bulk-Fill resins were developed to compensate for the disadvantages of polymerization shrinkage. Leprince et al.[11] said that more research is needed on whether to use it where a large occlusal force is required due to the low mechanical properties of most Bulk-Fill materials compared to existing composite resins. A study by Noh et al.[4] also reported that the physical properties of Bulk-Fill composite resins were inferior to those of traditional composite resins. On the other hand, regarding the polymerization depth according to the viscosity of the Bulk-Fill resin, a sufficient polymerization rate was reported for flowable Bulk-Fill resin at a depth of 4 mm[12], while there was controversy about packable Bulk-Fill resin[13,14].

Aguiar et al.[15] reported that the degree of polymerization of composite resin was affected by the size and shape of the photopolymerizer, the distance between the resin and the photopolymerizer, the intensity of the irradiated light, the irradiation time, the color and transparency of the resin, and the components of the resin thickness. Ahn et al.[8] reported that it is good to polymerize composite resins as close as possible, but clinically this cannot be satisfied in all cases, so it is necessary to increase the relative light intensity and irradiation time in order to increase the microhardness. And Bea et al.[16] found that the the manufacturer's recommended time of 20 seconds was not sufficient for the light-curing time of TheraCal Lc, a light-curing resin cement, under the polymerization of 1mm thick specimens at all cavity depths. Therefore, the thicker the thickness of the specimen, the more light irradiation time was required.

2. Objectives

Through these studies, it can be seen that existing composite resins are affected by various conditions during light polymerization, and depending on the conditions, manufacturer's recommended light curing time is insufficient. In addition, most studies on Bulk-Fill resins are comparative studies on shrinkage during polymerization between conventional composite resins and Bulk-Fill resins, and studies on light polymerization conditions of

Table 1. The List of Experiment Material

Name of material	Shade/type	Composition	Filler type	Filler content (vol%/wt%)	Manufacturer
Smart Dentin Replacement (SDR)	Universal /flowable	Modified UDMA, EBPDMA, TEGDMA	Ba-Al-F-B-silicate glass Average size 4.2 μ	44/68	Dentsply Sirona, USA
Tetric N-ceram Bulk-Fill	IVA /packable	Bis-GMA, UDMA	Ba-Al-silicate glass prepolymer filler 0.04～3 μ	55/77	Ivoclar-Vivadent, Liechtenstein

vol: volume, wt: Weight, UDMA: Urethane dimethacrylate, EBPDMA: EthoxylatedBisphenol-A Dimethacrylate, TEGDMA: Triethylene Glycol Dimethacrylate.

Bulk-Fill resins themselves are lacking. Therefore, this study selected Smart Dentin Replacement (SDR) as a flowable Bulk-Fill resin and Tetric N-ceram Bulk-Fill as a packable Bulk-Fill resin among Bulk-Fill resins most commonly used in clinical practice. Specimens were prepared by varying the curing time and distance of the two selected Bulk-Fill resins, and then the microhardness of the surface was compared and the curing time and distance that gave the optimum hardness were confirmed. This study was conducted to help more efficient treatment progress and satisfactory treatment results in dental clinical practice.

Materials and Methods

1. Research materials

In this study, SDR was used as a flowable Bulk-Fill resin, and Tetric N-ceram Bulk-Fill was used as a packable Bulk-Fill resin. The characteristics of research materials are as follows (Table 1).

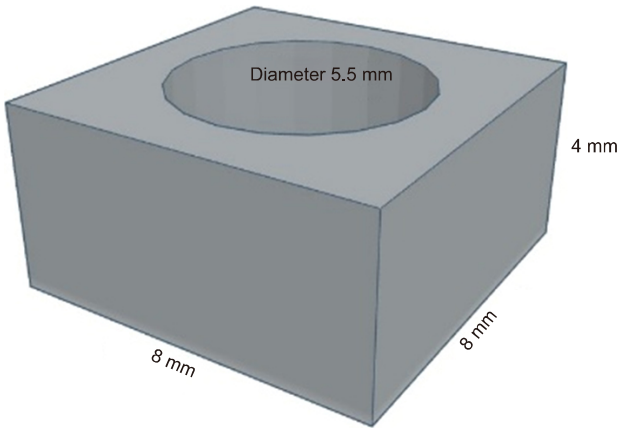

Fig. 1. 3 dimensional printer output.

2. Research design

1) Production of specimens

A total of 16 groups were classified according to the light curing time (10 seconds, 20 seconds, 40 seconds, 60 seconds) and the distance between the tip of the light curer and the material surface (0 mm, 2.0 mm, 4.0 mm, 6.0 mm) and each group had 10 specimens were produced. As for the light curing machine, a dental visible light curing machine was used (Noblesse, Max Dental Co., Korea). To fabricate the specimen, a plastic structure was printed using a 3D printer (LUGO Labs e-150, Lugo labs co., Korea). The plastic structure had an external dimension of 8 mm×8 mm×4 mm (width×length×height) and was manufactured in the form of a cylindrical hole with a diameter of 5.5 mm on the inner surface (Fig. 1). After injecting SDR and Tetric N-ceram Bulk-Fill into the plastic cuboid 3D printer output, respectively, a cover glass with a thickness of 0.15 mm was covered on top. The group with a polymerization distance of 0 mm was curing with light by placing the tip of the photopolymerizer in close contact with the cover glass, and the other groups were polymerized by varying the set distance and polymerization time on the cover glass. In order to accurately determine the polymerization distance, additional pillars were produced with a 3D printer and cured using the pillars (Fig. 2). In case of light curing time, 10 seconds and 20 seconds were used as machine's settings. For 40 seconds, the light was irradiated continuously twice with a setting of 20 seconds and for 60 seconds three times continuously with a setting of 20 seconds. All specimens were produced by one experimenter to minimize errors.

2) Measurement of surface microhardness

The surface of the fabricated specimen was not polished

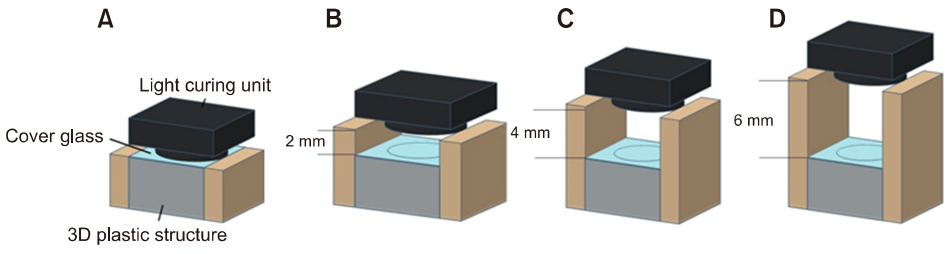

Fig. 2. Method of light-curing at different distances with specimen. Distance of 0 mm (A), 2 mm (B), 4 mm (C), 6 mm (D).

and after the specimen was pressed with a load of 100 gf for 10 seconds, the Vickers Hardness Number (Matsuzawa MMT-X, Matsuzawa co., Japan) was measured at a magnification of 400 times. Microhardness was measured in four directions, up, down, left, and right from the center of the specimen. To minimize errors, one experimenter measured the microhardness of all specimens.

3) Statistical analysts

The Kruskal-Wallis test was performed to evaluate the effect of the light curing time and the distance from the curing machine tip on the surface microhardness of the specimen, respectively, and the post-analysis was performed using the Mann-Whitney test. Post-hoc analysis was performed at a significance level of 0.0083 corrected by the Bonferroni method. The statistical data analysis program used SPSS 20.0 (SPSS Inc., Chicago, IL, USA).

Result

1. Measurement of surface microhardness of SDR

As a result of measuring the surface microhardness, the surface microhardness decreased as the distance increased in all time groups in the change in the distance from the polymerizer tip according to the light curing time, and there was a significant difference ($p < 0.05$, Table 2). As a result of the post-hoc analysis, it was found that the difference in microhardness between 4 mm and 6 mm was the largest in all groups. In particular, the biggest difference was found in 4 mm (35.07±2.02) and 6 mm (31.46±1.98) of the 10 second group of light curing, and similar results were obtained in the 20 second group. The results of 40 second and 60 second groups also showed the greatest change at 4 mm and 6 mm. However, in the 10 second group, there was no difference in microhardness between 0 mm (36.60±2.85) and 2 mm (37.64±1.49). There was also no difference between 2 mm (36.60±1.83) and 4 mm (36.13±0.97) in the 40 second group and between 0 mm (37.99±1.78) and 2 mm (37.92±1.96) in the 60 second group.

Even in the change of light curing time with respect to the distance from the polymerizer tip, the surface microhardness increased as the time increased in all distance groups, and the result was significant ($p < 0.05$, Table 3). As a result of the post-hoc analysis, the 0 mm group showed the greatest difference at 20 seconds (36.48±1.84) and 40 seconds (37.69±2.40) but in the 2 mm group, the greatest difference was observed at 10 seconds (37.64±

Table 2. Mean Value of Vickers Hardness Number with Increasing Distance from SDR Surface to Tip of Light-Curing Unit

Curing time	Distance				
	0 mm	2 mm	4 mm	6 mm	p-value
10 sec	36.60±2.85[a]	37.64±1.49[a]	35.07±2.02[b]	31.46±1.98[c]	<0.01
20 sec	36.48±1.84[a]	34.95±1.73[b]	33.20±1.45[c]	31.41±2.05[d]	<0.01
40 sec	37.69±2.40[a]	36.60±1.83[b]	36.13±0.97[b]	35.14±1.36[c]	<0.01
60 sec	37.99±1.78[a]	37.92±1.96[a]	37.77±1.45[a]	35.69±1.73[b]	<0.01

Values are presented as mean±standard deviation.
[a~d]When comparing within rows, different letters indicate significant differences by mann-whitney test as post-hoc test.

Table 3. Mean Value of Vickers Hardness Number of SDR with Increasing Light-Curing Time

Distance	Curing time				
	10 sec	20 sec	40 sec	60 sec	p-value
0 mm	36.60±2.85[a]	36.48±1.84[a]	37.69±2.40[b]	37.99±1.78[b]	<0.01
2 mm	37.64±1.49[a]	34.95±1.73[b]	36.60±1.83[c]	37.92±1.96[a]	<0.01
4 mm	35.07±2.02[a]	33.20±1.45[b]	36.13±0.97[c]	37.77±1.45[d]	<0.01
6 mm	31.46±1.98[a]	31.41±2.05[a]	35.14±1.36[b]	35.69±1.73[b]	<0.01

Values are presented as mean±standard deviation.
[a~d]When comparing within rows, different letters indicate significant differences by mann-whitney test as post-hoc test.

1.49) and 20 seconds (34.95±1.73). In the 4 mm group, the greatest change was seen at 20 seconds (33.20±1.45) and 40 seconds (36.13±0.97), and the greatest difference was also seen at 20 seconds (31.41±2.05) and 40 seconds (35.14±1.36) at 6 mm. Therefore, in most groups, the greatest change was observed at 20 seconds and 40 seconds. However, in the 0 mm group, there was no significant difference between 10 seconds (36.60±2.85) and 20 seconds (36.48±1.84), and between 40 seconds (37.69±2.40) and 60 seconds (37.99±1.78), respectively. Also, in the 6 mm group, there was no significant difference between 10 seconds (31.46±1.98) and 20 seconds (31.41±2.05), and between 40 seconds (35.14±1.36) and 60 seconds (35.69±1.73).

2. Measurement of surface microhardness of Tetric N-ceram

In the change in the distance from the polymerizer tip to the light curing time, the microhardness decreased as the distance increased in the 10 second and 40 second groups, and the result was significant ($p < 0.05$, Table 4), but in the case of the 20 second and 60 second groups, the change in the distance from the curing machine tip was not significant with respect to the light curing time ($p > 0.05$, Table 4). As a result of the post-hoc analysis, the 10-second light curing group showed the greatest difference between 0 mm (41.07±3.05) and 2 mm (39.70±2.26), and In the 40 second group, the biggest difference was found at 2 mm (46.18±6.60) and 4 mm (47.43±1.27).

In the change of time for the distance from the light curer tip, the surface microhardness increased as time increased in all distance groups, and the result was significant ($p < 0.05$, Table 5). As a result of the post-hoc analysis, it was confirmed that the change between 20 and 40 seconds was the greatest in most groups. The 0 mm group showed the greatest change at 20 seconds (42.70±5.00) and 40 seconds (48.44±1.45), and the 2 mm group also showed the greatest change at 20 seconds (42.70±2.70) and 40 seconds (46.18±6.60). In the 4 mm group, the biggest difference was shown at 10 seconds (38.56±2.00) and 20 seconds (43.63±1.87), but in the 6 mm group, the greatest difference was shown at 20 seconds (42.95±1.46) and 40 seconds (47.02±2.24).

Discussion

1. Interpretation

Dental caries is irreversible damage to tooth, and

Table 4. Mean Value of Vickers Hardness Number with Increasing Distance from Tetric N-Ceram Surface to Tip of Light-Curing Unit

Curing time	Distance				
	0 mm	2 mm	4 mm	6 mm	p-value
10 sec	41.07±3.05[a]	39.70±2.26[b]	38.56±2.00[b]	38.92±3.28[b]	<0.01
20 sec	42.70±5.00[a]	42.70±2.70[a]	43.63±1.87[a]	42.95±1.46[a]	0.051
40 sec	48.44±1.45[a]	46.18±6.60[a]	47.43±1.27[b]	47.02±2.24[b]	<0.01
60 sec	48.50±2.04[a]	48.22±1.21[a]	47.82±1.33[a]	48.03±1.54[a]	0.065

Values are presented as mean±standard deviation.
[a,b]When comparing within rows, different letters indicate significant differences by mann-whitney test as post-hoc test.

Table 5. Mean Value of Vickers Hardness Number of Tetric N-Ceram with Increasing Light-Curing Time

Distance	Curing time				
	10 sec	20 sec	40 sec	60 sec	p-value
0 mm	41.07±3.05[a]	42.70±5.00[b]	48.44±1.45[c]	48.50±2.04[c]	<0.01
2 mm	39.70±2.26[a]	42.70±2.70[b]	46.18±6.60[c]	48.22±1.21[c]	<0.01
4 mm	38.56±2.00[a]	43.63±1.87[b]	47.43±1.27[c]	47.82±1.33[c]	<0.01
6 mm	38.92±3.28[a]	42.95±1.46[b]	47.02±2.24[c]	48.03±1.54[d]	<0.01

Values are presented as mean±standard deviation.
[a~d]When comparing within rows, different letters indicate significant differences by mann-whitney test as post-hoc test.

composite resins with sufficient compressive strength, indirect tensile strength, and modulus of elasticity have been used to restore damaged tooth to their original state[17]. Resin undergoes polymerization shrinkage[2], and Bulk-Fill resin was developed to overcome this[4,11]. Although these Bulk-Fill resins have been able to escape the disadvantage of polymerization shrinkage, there is a lack of research on polymerization time or polymerization distance that can have sufficient physical properties.

There are direct methods and indirect methods to determine the physical properties of resin[18,19]. A direct method is to measure the amount of monomer, and one of the indirect methods is a comparison using microhardness[19,20]. Microhardness is a physical property that represents the characteristics of a material and represents the magnitude of the force that a material resists permanent deformation that occurs when an external force is applied to a minute part[21]. Therefore, in this study, Vickers Hardness Number was used to measure the physical properties of Bulk-Fill resin.

As for the polymerization time, Bae et al.[16] study showed that, in the case of TheraCal LC, the hardness was low at all cavity depths with the 20 second light irradiation time recommended by the manufacturer. and Cho et al.[22] study also reported that the surface hardness of the composite resin was higher with a polymerization time of 20 seconds or longer than that of a specimen with a polymerization time of 20 seconds. Through the above studies, it was found that the polymerization time of 20 seconds commonly used in clinical practice is insufficient for sufficient hardness of the resin. SDR and Tetric N-Ceram, the Bulk-Fill resins used in the experiment, also indicated only a light curing time of 20 seconds or more. Therefore, in this study, based on the clinical light curing time of 20 seconds, 1/2 time for 10 seconds, 2 times for 40 seconds, and 3 times for 60 seconds were conducted. Based on Lokade et al.[23] research, the distance from the light curer tip was calculated as the maximum curing distance of 6 mm, which is the average value of the distance to the cusp. The distance was divided by dividing the distance between the minimum polymerization distance of 0 mm and the maximum polymerization distance of 6 mm in units of 2 mm.

In the change of distance for light curing time of SDR, as the distance increased, the surface microhardness decreased in all time groups, and the result was significant ($p < 0.05$). In particular, the difference in microhardness between 4 mm and 6 mm was large in all time groups. At a light curing time of 10 seconds, the surface microhardness increased at 2 mm rather than at 0 mm, but there was no significant difference as a result of post-mortem analysis. In the change of time for the distance from the SDR light curer tip, the surface microhardness increased as the light irradiation time increased in the distance group, and the result was significant ($p < 0.05$, Table 3), and In all distance groups, the difference between the 20 second and 40 second groups was the largest. However, in comparison between 10 seconds and 60 seconds at 2 mm, the two groups showed similar hardness, and the hardness of the 10 second group at 4 mm was lower than that of the 20 second group. This is thought to be caused by an error due to an unknown variable during specimen production. In Bae et al.[16] study, the diameter of the hole in the 3D printer was similar to the tip of the light curing machine, so it was placed in the center of the specimen more than the case with a pillar next to it, and a high intensity of light was irradiated, or an error could occur due to the movement of the pillar during specimen production.

Regarding the change in distance for the light curing time of Tetric N-ceram, the change in surface microhardness was not significant as the distance from the light curing tip increased in the 20 and 60 second groups ($p > 0.05$). For that reason, the error of the specimen mentioned above is expected as a result. Hwang et al.[24] report also stated that there is a possibility that the work hardened layer, which is not the original hardness value of the specimen, can be measured in the hardness value during specimen production. However, in the 10 second and 40 second groups, the surface microhardness decreased as the distance from the light curer tip increased, and the result was significant ($p < 0.05$). In the 10 second group, the 0 mm and 2 mm groups showed the greatest difference, and in the 40 second group, the 2 mm and 4 mm groups had the greatest difference. In the time change of the distance from the tip of the light curing machine of Tetric N-ceram, as the light curing time increased, the surface microhardness significantly increased. In the study using the bulk-base resin, a

significant difference in surface microhardness change was found from 4 mm in the case of the high flow bulk resin. In addition, it was reported that there was a significant change in surface microhardness between 0 mm and 2 mm of Medium Flow Bulk resin, but no significant change in surface microhardness was found between 2 mm and 3 mm, and 3 mm and 4 mm[21]. This shows similar results when compared to SDR, a flowable Bulk-Fill resin used in this study, and Tetric N-ceram resin, a packable Bulk-Fill resin. In a study by Diab et al.[25], the surface microhardness of Bulk-Fill resin decreased as the polymerization distance increased from a light curing time of 20 seconds. Although the decrease was not significant, it was reported that there was a significant decrease in surface microhardness from 8 mm. Also, through Hasanain et al.[26] research, although there are differences depending on the material of the Bulk-Fill resin, it is recommended to perform light polymerization at the closest distance as possible because the distance from light polymerization and the depth of the material affect the surface microhardness. Summarizing the above research results, it is thought that SDR requires 40 seconds of light irradiation time at a distance of 4 mm or less, and more sufficient light irradiation time at a distance of 4 mm or more. Tetric N-ceram's hardness increased up to 40 seconds of light irradiation time at most distances, and it is thought that more sufficient light irradiation time is required as the distance increases.

2. Limitations and suggestion

Since this study was not applied to the dental cavity in the human body, the results of the study may appear different from reality. AlShaafi et al.[27] study on the effects of mold type and diameter size on the degree of polymerization of resin bases reported that the material and diameter size affect the degree of photopolymerization. Therefore, in future studies, it is considered necessary to study the difference in degree of polymerization according to the size of the tooth cavity in a situation similar to the oral environment.

Notes

Conflict of interest

No potential conflict of interest relevant to this article was reported.

Ethical approval

This article does not require for IRB screening because human origin is not used.

Author contributions

Conceptualization: Hyung-Min Kim and Do-Seon Lim. Data acquisition: Hyung-Min Kim. Formal analysis: Hyung-Min Kim, Moon-Jin Jeong, Hee-Jung Lim, and Do-Seon Lim. Supervision: Moon-Jin Jeong, Hee-Jung Lim, and Do-Seon Lim. Writing-original draft: Hyung-Min Kim. Writing-review & editing: Hyung-Min Kim, Moon-Jin Jeong, Hee-Jung Lim, and Do-Seon Lim.

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None.

Data availability

The supporting data of this study are available from the corresponding author upon reasonable request.

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