Microhardness and degree of conversion of three bulk fill resin composites in different depth.

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Abstract

Objective: The aims of the study were to evaluate the microhardness and the degree of conversion of three bulk-fill resin composites.

Materials and Methods: Three bulk-fill resin composites (1. Filtektm Bulk Fill ,3M ESPE (Filtek) 2. Sonic Filltm , Kerr (Sonic) 3. Tetric® N-Ceram Bulk Fill, Ivoclar- Vivadent (Tetric)) were investigated using Vickers hardness test at three depths of cure (2mm/4mm/5mm). After polymerization, the samples were kept in the distilled water under light protection environment for 24 hours prior to the test. Then, the degree of conversion was measured by Fourier transform infrared spectroscopy (FTIR).

Results: The Vickers hardness values of the top and the bottom surfaces of all specimens were as follows: at 2 mm depth of cure, Sonic 79.95/71.45 Tetric 58.05/52.72 and Filtek 64.84/61.00. At 4 mm depth of cure, Sonic 76.17/66.64, Tetric 57.61/53.74 and Filtek 65.04/62.33. At 5mm depth of cure, Sonic 77.25/42.16, Tetric 57.91/42.96 and Filtek 65.25/50.75. On top surfaces, there were larger degree of conversion in Sonic than Tetric and Filtek respectively. However, at the other depths, Tetric showed larger degree of conversion than Sonic and Filtek respectively

Conclusion: All groups displayed unsatisfactory polymerization and microhardness at 5-millimeter depth. Different bulk-fill resin composite exhibited different reduction pattern of microhardness and degree of conversion.

Keyword: *bulk-fill resin composite, Vickers Microhardness, Degree of conversion*

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Introduction

Resin composite has been widely used as an alternative to amalgam. The material provides satisfactory performance in terms of cosmetic dentistry. 1 However, polymerization shrinkage has been the utmost concern when working with resin composite. 2 To overcome the problem, the incremental technique was recommended to achieve the longevity of resin composite restorations. 3 Through the incremental technique, the material is filled layer by layer to reduce the polymerization shrinkage and the C-factor which causes the restoration to have microleakage at the margin or microcrack of the enamel. 4,5 The incremental technique is time consuming but cannot guarantee the marginal seal. It was reported that the risk of post-operative sensitivity was not affected by adhesive mode or the filling technique. 6 Moreover, a study reported that incremental technique was worse than bulk technique at cementum margin. 7 Recently bulk-fill resin composite was introduced into the market. The cavity can be filled with just one bulk up to 4 or 5 millimeters as claimed by the manufacturers. According to the developers, their new monomer is able to provide promising properties such as decreasing the shrinkage, improving the mechanicalphysical properties, improving the degree of conversion. 8 It seems that this material might be a remedy for polymerization shrinkage. Microhardness is commonly used as the method for verifying the mechanical property of resin composite based materials. 9,10 The degree of conversion is a method used to quantify the monomer polymerization in the polymer. The higher degree of conversion signifies a better quality of polymerization. 1,11

The aims of this study were to investigate the degree of conversion and physical property (Vicker 's microhardness) of investigate three bulk-fill resin composites and to compare the degree of conversion and physical property (Vicker 's microhardness) after being photo polymerized at three different depths (2,4, and 5 mm).

Materials and Methods

Specimen preparation

Thirty specimens with 10-mm in diameter were prepared for three bulk-fill resin composites. The materials used in this study were listed in Table 1. Each group of material was filled into silicone molds at three depths 2, 4, and 5 mm and covered with a glass slide on top. Then Elipar™ S10 LED Curing Light (3M ESPE, USA) was used to cure the material for 40 seconds. Through this means, the oxygen inhibited layer was not affected at the surface and the distance from the light tip to the resin surface was controlled. The power density of light curing unit was assessed using a hand-held radiometer. The top surface of all specimens was indented and marked. After complete curing, specimens were then removed from molds and stored in distilled water in a light protection box at room temperature for 24 hours before testing.

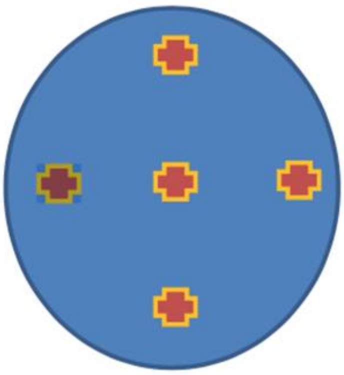
Material	Filler	Component	Light curing protocol	Depth of cure

Tetric® N-Ceram Bulk Fill (Ivoclar Vivadent, Germany)	61% (vol.) 79 – 81% by wt	Resin : Bis-GMA, Bis-EMA and UDMA Filler : Ba-Al-Si glass , prepolymerized filler, Mixed oxide	> 1,000mW/cm^2 , 10 sec.	4 mm.
SonicFill™ Bulk Fill (Kerr, USA)	83.5 % wt	Resin : Bis-GMA, TEGDMA, EBPDMA Filler: SiO_2, Glass, oxide	> 550 mW/cm^2, 20 sec	5 mm.
Filtek™ Bulk Fill (3M ESPE,USA)	76.5% by wt (58.4% by vol.)	Resin : AUDMA, UDMA, AFM Structure and 1, 12-dodecane-DMA Filler : Zr , Zr-Si glass, YbF_3	> 1,000mW/cm^2 , 20 sec.	5 mm.

Table 1 Lists of material used in this study

Vicker Microhardness Test

Microhardness tester (Microhardness Tester model FM-700e, Future Tech) was used to test the hardness of specimens. The test were performed at a load of 300g with 15 seconds dwelling time on top and bottom surface of specimens. Each surface was tested by the diamond tip of hardness tester for 5 points. The mean values from 5 points were recorded as the hardness of each surface as shown in Figure 1.



Fi esting points

Degree of Conversion

The degree of conversion was measured using Fourier transform infrared spectroscopy FTIR (Model Spectrum 1, Perkin Elmer USA). Degree of conversion was measured by assessing the variation in the ratio of the absorbance intensities of aliphatic C=C peak. The FTIR spectrometer measured aliphatic absorption peak at 1638 cm-1 and Aromatic absorption peak at 1610 cm-1. Thus, the degree of conversion was calculated. For the degree of conversion, the top surface was collected from the 2 mm group as the representative of maximum cured surface. The bottom surface of each group was evaluated. Since the top surface of each group was exposed to the light at the same level t. Therefore, using one group as the reference for the maximum irradiation is deemed to be adequate.

Statistical Analysis

The Shapiro-Wilk test was used to evaluate the distribution of the data. The Two-way ANOVA was used for detecting the differences among experimental groups. The Scheffe multiple comparisons test was used as post hoc test.

Results

Vickers Hardness

The summary of the hardness results is shown in Figure 2. FiltekTM Bulk Fill and Tetric® N-Ceram Bulk Fill exhibited no significantly different from the top surface to 4 mm-surface. SonicFillTM exhibited significantly difference value in every depth.

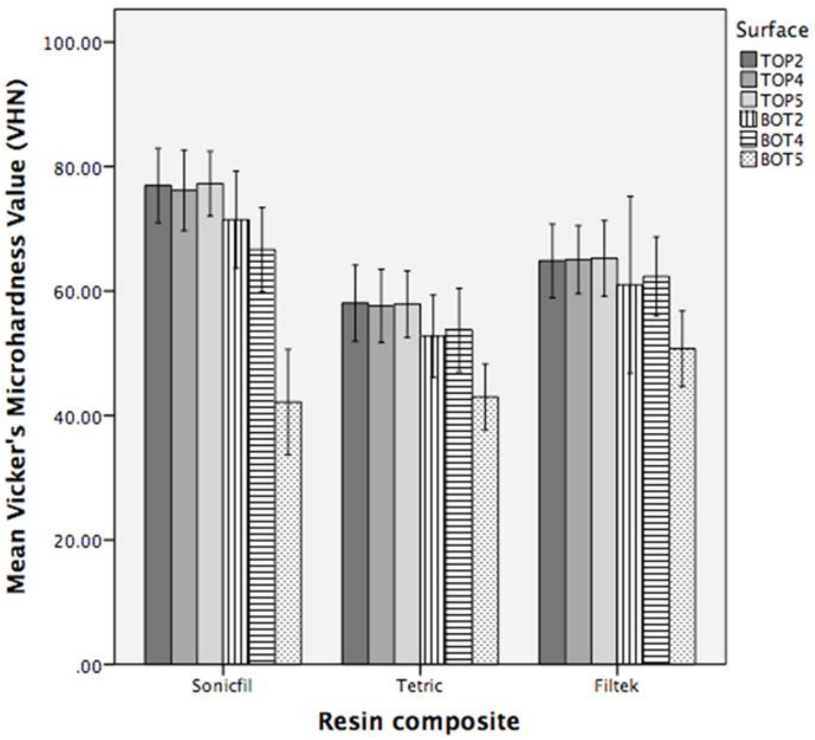


Figure 2 Mean Vicker's microhardness result

Degree of Conversion

The average degree of conversion is shown in Figure3. Tetric® N-Ceram Bulk Fill exhibited the highest degree of conversion from 2 mm. up to 5 mm. depth. Whereas SonicFillTM delivered the highest degree of conversion at top surface and then dropped dramatically. At 5 mm. depth, SonicFillTM showed the lowest degree of conversion. Two-way ANOVA revealed no significantly difference values for Tetric® N-Ceram Bulk Fill at top surface, 2 mm. depth and SonicFillTM at top surface.

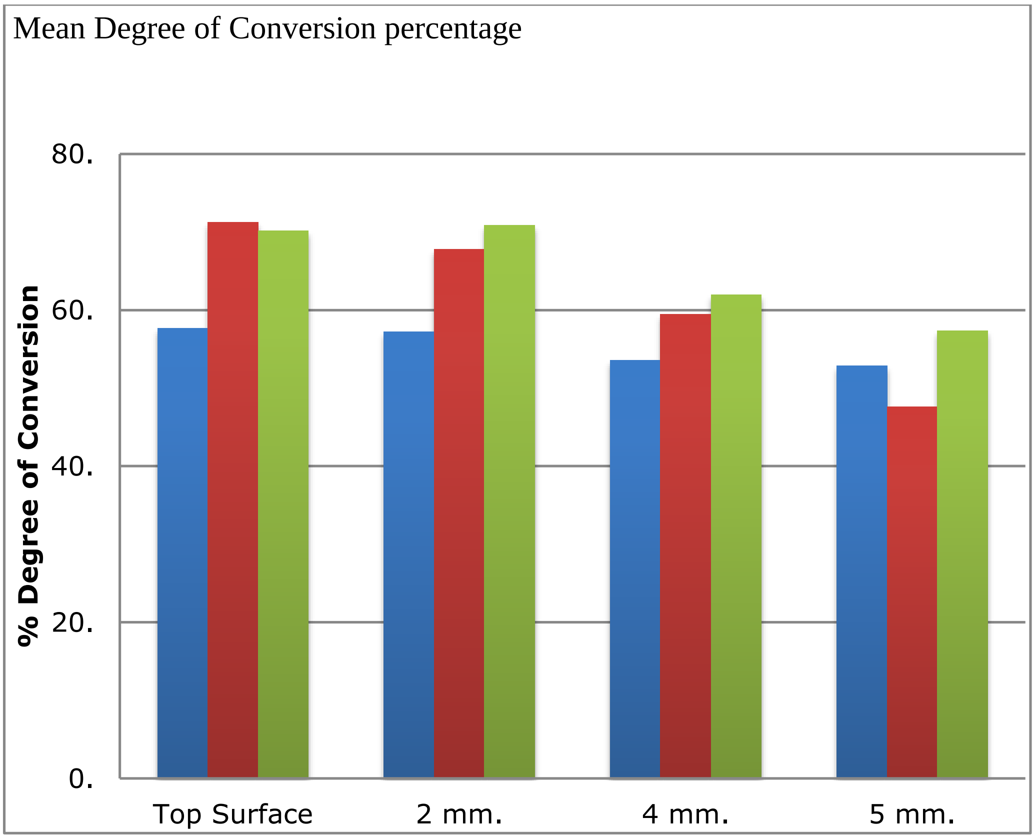


Figure 3 Mean percentage of degree of conversion

Discussion

Although recently launched, bulk-fill resin composite has gained attention from many dentists. A number of the studies investigated the material in many aspects. 12,13,14 The top surface of every tested materials in this study showed the highest degree of conversion and microhardness values. This result was unforeseen because the top surface of each group was close to the light source compared to other depths. It was reported that the distance of the light tip from resin composite could reduce the microhardness and the degree of conversion of the resin composites. 15 However, there were still some significant differences of microhardness and degree of conversion among three resin composites used in this study. This is because there are many factors affecting the hardness and degree of conversion such as the resin matrix, filler system and light source. 16 At top surface, SonicFillTM exhibited the highest microhardness and degree of conversion. From Table 1, the three resin composites are different in term of resin matrix and filler. SonicFillTM achieved the highest microhardness at the top surface. It has been proved by researchers that higher filler loading provides better mechanical and physical properties. Lohbauer et al. concluded that increasing the filler loading could enhance elastic modulus and mechaniphysical properties.17 However, the microhardness drops dramatically when the depth exceeded 4 mm. The fact that the differences of hardness between 4 mm and 5 mm were statistically significant , it is indicated that the material could not be used in a bulk exceeding more than 4 mm. Another point that should be concerned was the degree of conversion dropped dramatically when the thickness of materials increased. This study revealed that the high value of microhardness did not correlate with the high degree of conversion. The increasing size, volume and type of the filler also affected the increasing of microhardness value.18 The filler particles are added into resin composite to provide improvement of mechanical and physical properties . Also, reduction of volumetric shrinkage and thermal expansion or contraction, decreased water sorption and ease of manipulation.19

The results showed that Tetric® N-Ceram Bulk Fill displayed the highest value of degree of conversion among all specimens but the least microhardness value except at 5 mm depth. It was shown in table 1 that Tetric® N-Cerem Bulk Fill has a different filler system from the other two resin composites. The pre-polymerized fillers may cause the differences in mechanical and physical properties. Although the main composition of the fillers is a silicate glass, Ferracane et al. reported that the pre-polymerized particles caused the reduction in fracture toughness. 4 Although the material did not perform well in terms of mirohardness, Tetric® N-Ceram Bulk Fill exhibited the best degree of conversion in any depth compared with other two resin composites. It has been well established that the degree of conversion can be affected by type of resin matrix and fillers, Tetric® N-Ceram Bulk Fill, which contains Bis-EMA, providing higher degree of conversion compared to Bis–GMA. 13 This can be one of the factors attributed to the higher degree of monomer conversion 20. However, Bis-EMA was reported to be eluted from the bond interface more than Bis-GMA due to hydrolysis. 21

SonicFillTM displayed highly reduction of the degree of conversion and microhardness at 5 mm. depth, the degree of conversion has decreased to 47.633% and the microhardness value has reduced to 42.16 VHN. This result was significantly different when compared to the top surface. It may be caused by the increased viscosity of the resin matrix during the polymerization process. One study has shown the correlation of the shrinkage stress and the degree of conversion. 22 At 5-mm-depth group, the shrinkage stress and viscosity may increase and interfere with the polymerization of the resin matrix. This characteristic could affect the handling property of this material. FiltekTM Bulk Fill exhibited the lowest degree of conversion except at 5 mm. However, the change of the degree of conversion from 2 mm to 5 mm was not extremely high as shown in figure 2. The microhardness value of FiltekTM Bulk Fill is higher than Tetric® N-Ceram Bulk Fill in any depth and higher than SonicFillTM at 5 mm. From table 1, FiltekTM Bulk Fill has the different filler system from the others as it contains zirconia. A study reported that reinforcing the resin composite with the optimum amount of zirconia filler can increase the fracture toughness of the material. However, the reinforcement could reduce the degree of conversion of tested resin composite. 23 It is possible that the zirconia filler system provided the higher hardness for FiltekTM Bulk Fill However, it could affect the degree of conversion of the material.

Although the tested materials showed different result in the two mechanical and physical aspects, this study found clear evidence that the 3 bulk-fill resin composites should not be used when the depth is more than 4 mm. The reduction of microhardness and degree of conversion at 5 mm. indicated the depth limitation of the tested materials. From this study, none of the tested materials can be used at a bulk of 5 mm in thickness. The lower degree of conversion may increase the chance of free monomer leaking into the body. In addition, most resin composites contain monomer, which is the derivative of Bisphenol A (BPA). BPA was reported to be the endocrine disruptor with toxicity potential. 24 It was also concluded that bulk-fill resin composite is not suitable to be used when the degree of conversion of the material is less than 80 percent of the top surface. (23) The LED light curing unit was used throughout the study. However, the high intensity of LED light curing unit may affect the mechanical and physical properties of the tested materials. For example, the high intensity energy can cause more polymerization and degree of conversion. 25 In a recent study, SonicFillTM was cured with low intensity light. The material showed less surface loss in the study. 26 Since we did not compare the light source, therefore, the effect of the light source may be underestimated in this study.

Conclusion

All groups displayed unsatisfactory polymerization and microhardness at 5-millimeter depth. Different bulk-fill resin composite exhibited different reduction pattern of microhardness and degree of conversion.

The result from this study indicated that the bulk-fill resin composites should not be used when the thickness was more than 4 mm. However, the materials seemed to perform well in the different condition from one to another. The long term clinical data should be collected to provide the accurate information of the materials. Also, other physical properties such as elastic modulus, wear resistance, color matching should be reported to provide more information for the clinicians in order to have a better understanding of the available materials.

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ค่าความแข็งระดับจุลภาคและ ปริมาณการเกิดพอลิเมอไรเซชันของวัสดุ บัลค์ฟิลเรซินคอมโพสิทที่ระดับความลึกต่างกัน

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บทคัดย่อ

วัตถุประสงค์: เพื่อศึกษาค่าความแข็งผิวระดับจุลภาคและปริมาณการเกิดพอลิเมอไรเซชันของ วัสดุเรซินคอมโพสิต ชนิดบัลค์ฟิลล์

วัสดุอุปกรณ์และวิธีการ: ทำการศึกษาเปรียบเทียบเรซินคอมโพสิตสามชนิด (1. Filtek™ Bulk-Fill, 3M ESPE (Filtek) 2. Sonic-Fill™,Kerr (Sonic) 3. Tetric® N-Ceram Bulk Fill, Ivoclar Vivadent (Tetric)) หลังจากบ่มด้วยแสงที่ ระดับความลึกต่างๆคือ 2, 4 และ 5 มิลลิเมตร โดยทำการทดสอบค่าความแข็งระดับจุลภาควิคเกอร์ และปริมาณการเกิดพอลิเมไรเซชัน ด้วยเครื่องฟลูเรียร์ทรานส์ฟอร์มอินฟราเรดสเปคโทรมิเตอร์ (FTIR) โดยทำการทดสอบหลังจากตัวอย่างได้ทำการบ่มด้วยแสงแล้วเก็บไว้ในน้ำกลั่นและกล่องทึบแสงเป็นเวลา 24 ชั่วโมง

ผลการทดลอง : จากการทดสอบค่าความแข็งผิว ระดับจุลภาควิคเกอร์ ผิวบน/ผิวล่าง ได้ค่าดังนี้ ที่ชิ้นทดสอบความหนา 2 มิลลิเมตร พบว่ากลุ่ม Sonic ได้ค่า 79.95/71.45 กลุ่ม Tetric ได้ค่า 58.05/52.72 กลุ่ม Filtek ได้ค่า 64.84/61.00 สำหรับชิ้นทดสอบที่ความหนา 4 มิลลิเมตรพบว่ากลุ่ม Sonic ได้ค่า 76.17/66.64 กลุ่ม Tetric ได้ค่า 57.61/53.74 กลุ่ม Filtek ได้ค่า 65.04/62.33 และในชิ้นทดสอบที่ความหนา 5มิลลิเมตรพบว่ากลุ่ม Sonic ได้ค่า 77.25/42.16 กลุ่ม Tetric 57.91/42.96 กลุ่ม Filtek 65.25/50.75 ผลความแตกต่างของปริมาณการเกิดพอลิเมอไรเซชัน ในทั้ง3กลุ่มตัวอย่างเป็นดังนี้ คือในระดับผิวบนสุดพบว่า Sonic > Tetric > Filtek ส่วนในระดับความลึกอื่นๆจะเป็น Tetric > Sonic > Filtek โดยมีค่าปริมาณการเกิดพอลิเมอไรเซชันตั้งแต่ 47.623 ถึง 71.226 %

สรุปผลการทดลอง: ทุกกลุ่มตัวอย่างแสดงค่าความแข็งผิวและค่าปริมาณการเกิดพอลิเมอไรเซชันที่ต่ำกว่าเกณฑ์ที่เหมาะสมต่อการใช้งานที่ระดับ ความลึก 5 มิลลิเมตร นอกจากนี้เรซินคอมโพสิตต่างชนิดกันยังมีอัตราการลดลงของความแข็งและค่าปริมาณ การเกิดพอลิเมอไรเซชันที่ต่างกัน

**ภาควิชาทันตกรรมอนุรักษ์และทันตกรรมประดิษฐ์ คณะทันตแพทยศาสตร์ มหาวิทยาลัยศรีนครินทรวิโรฒ*

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