Original Article

The Effect of Clear Silicone Barrier and Polymerizing Depth on Hardness of Light-Polymerized Composite Resin

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Abstract

The objective of this study was to determine the effect of silicone thickness and the polymerizing depth of light-polymerized composite resin on the Knoop Hardness Number and the percentage of bottom-top Knoop Hardness Number of the resin. There were 4 groups of composite resin specimen with different thickness of clear silicone (0.0, 2.0, 4.0, and 6.0 mm). There are also 4 subgroups in each group with different polymerizing depth (0.25, 0.2, 0.4, and 6.0 mm). Ten specimens were made for each subgroup. Therefore, there were 160 specimens altogether. Knoop Hardness Numbers and percentage of bottom-top Knoop Hardness Numbers of the composite resin were measured and analyzed using a Two-way analysis of variance, and Tukey's HSD test ($p < 0.01$). There were significant differences ($p < 0.01$) among mean Knoop Hardness Numbers and percentage of bottom-top Knoop Hardness Numbers in all groups. Only 3 data points not less than 80 % were detected. In conclusion, the thickness of clear silicone of no more than 4.0 mm and the polymerizing depth of no more than 2.0 mm were used for adequate polymerization.

Key words: Composite resin; Knoop Hardness Number; Silicone barrier

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Introduction

Composite resins are some of the most broadly-used materials in restorative dentistry because of esthetic concerns by patients, improvements in their compositions and applications, and simplification of bonding systems.[1-4] It is noted that incomplete polymerization leads to many undesirable results, including reduced mechanical properties,[5] decreased bond strengths,[6] increased wear,[7] the marginal deterioration of the restoration,[8] and reduced biocompatibility.[9] Focusing on mechanical characteristics of composite resin materials, incomplete polymerization also affects, for example, strength, stiffness,[10] hardness,[11] and wear resistance.[12] There are several factors which are influence in polymerizing process of light-polymerized composite resin material, for example, distance between the light source and the target material, exposure time, and intensity of light source.[13-22]

Among several techniques to detect the degree of conversion of composite resins, Fourier Transformation Infrared Spectroscopy (FTIR) has been shown as a powerful and reliable technique.[23-28] To simplify the measurements, various indirect methods have been described in the literature.[29-31] These include changes in optical translucency,[29] scraping,[30] resin leaching[26] and hardness measurements.[31] The Knoop Hardness test has been proved to be a suitable method for the hardness measurement of composite resins. Because it has been reported a good correlation between the Knoop Hardness Number (KHN) and degree of conversion.[30,32] Moreover, it has been shown that Knoop Hardness test correlates well with FTIR.[26]

The depth of polymerization obtained, according to ISO 4049:2000, obviously depended on a lower degree of polymerization corresponding to an indentation hardness of 80 % of the irradiated surface.[33] However, to define the depth of polymerization based on bottom and top hardness measurements, the routine is to find out a minimum value of the ratio of bottom-top hardness so as to indicate the bottom surface for adequate polymerization. Hence, the ratio values of 0.80 and 0.85 (or 80 % and 85 % of the hardness of the irradiated surface respectively) have frequently been used.[34-35] Apart from that, Watt *et al*[36] defined an adequate polymerizing depth of a composite resin material as the depth where its hardness value corresponds to 80 % of the hardness of the irradiated surface. For measurement, it was clear that the indentations determined at the top of specimens were exactly at the top hardness data. But in some research the measurement was only at the side of specimens, and the minimum depth from the top surface was used as the top hardness data.[37-40]

Currently, to restore any teeth with composite resin, there are 3 techniques which are direct, direct-indirect, and indirect techniques. Most dentists use direct and direct-indirect techniques to improve bonding more than the indirect technique. In a tooth with extensive structural damage, the direct technique takes the extensive chair time and is difficult to obtain accurate contour and occlusal anatomy. Therefore, light-polymerized composite resin with the direct and direct-indirect techniques can be used by replication of an anatomically contoured waxing with a transparent index.

The bite registration Memosil 2 on A-silicone base is versatile and can be used from prosthetics to conservative dentistry to implantology and orthodontics.[47] It has a high final hardness and can be taken out of the mouth without risk of breakage. The automatic mixing system saves time and reduces the risk of failure. The silicone is a transparent A-silicone for special indications. Light-curing is possible for filling and fixative materials through placeholders with the silicone.[41] There are two researches which reported the effect of the silicone thickness on microhardness of dual-polymerized composite resin provisional restoration.[42-43] However, there is no report of light-polymerized composite resin. Therefore, this silicone may also be used as a clear silicone index for light-polymerized composite resin. The present study aimed to investigate the effects of two independent variables: the thickness of clear silicone barrier and the polymerizing depth of light-polymerized composite resin. These variables may affect two dependent variables: the Knoop Hardness Number and the percentage of bottom-top Knoop hardness. The null hypotheses tested were that the clear silicone thickness and the polymerizing depth of light-polymerized composite resin have no effects on (1) the KHN of the composite resin and (2) the percentage of bottom-top Knoop hardness number of light-polymerized composite resin.

Materials and Methods

A microhybrid composite resin (Clearfil shade A3.5; Kuraray Medical Inc, Japan) and the clear silicone (Memosil 2; Heraeus Kulzer GmbH, Germany) (Table 1) were prepared with the L&P mold (Patent No. 1001000915) (Fig. 1) and the L&P method (Patent No. 1103000789). A light-polymerizing unit (Pekalux; Heraeus Kulzer GmbH, Germany) was applied to the L&P mold. KHN was determined with a digital microhardness instrument (MHT10; Anton Paar USA Inc, USA).

Specimen preparation

The L&P mold is a rectangle metal mold with a chamber. A typical chamber in the center of the metal mold was divided into 3 compartments: the upper, the middle, and the lower compartment (Fig. 2). The upper compartment, 10 mm in height, was used for holding a light-tip guide of the light-polymerizing unit. The middle compartment, 6 mm in height, was used for holding silicone barrier while polymerizing a composite resin specimen. The lower compartment, 8 mm in height, was used for 3 reasons. The first reason was for fabricating the composite resin specimen. The second reason was for fabricating silicone barrier. The third reason was for fixing the composite resin specimen during the measurement of the Knoop hardness. The light-tip diameter of the light-polymerizing unit was 10.0 mm which matched the dimension of the upper compartment. In the middle compartment, the 6.0 mm fixed distance between the light-tip to the composite resin specimen was used for all 160 specimens. The 6.0 mm distance was filled with either air or the silicone or a combination between air and silicone.

Table 1 Products used for testing

Product	Manufacturer	Shade
Memosil 2	Heraeus Kulzer GmbH, Germany	clear
Clearfil AP-X	Kuraray Medical Inc., Japan	A3.5

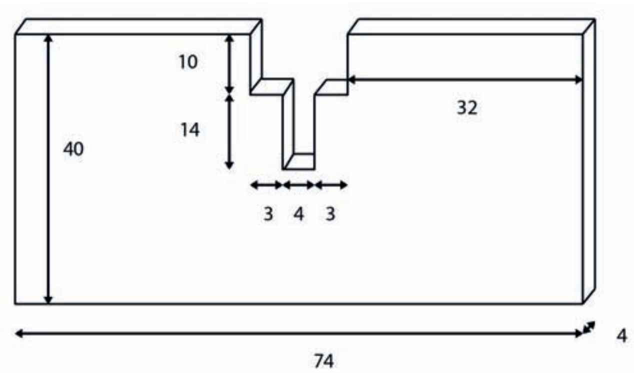

Figure 1 L&P Mold

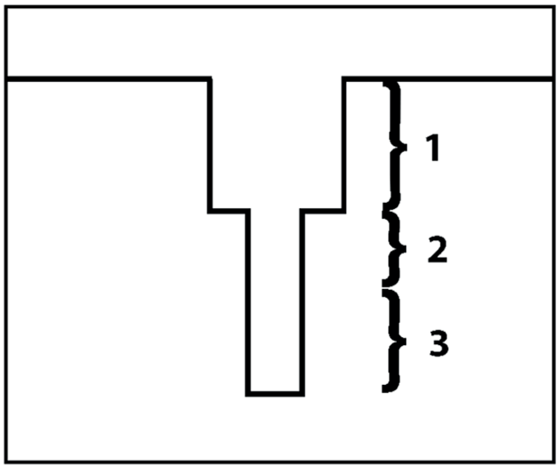

Figure 2 *Close up L&P mold chamber: number 1, 2 and 3 represent the upper, middle and lower part*

There were 2 steps in specimen preparation. For the first step, 3 silicone barriers were made by injecting material into the lower compartment space between the 2 opaque glass slabs. There were 3 heights: 2.0, 4.0, and 6.0 mm. For the second step, 4 groups of composite resin specimen were made by different heights of silicone barrier. There were 4 heights of silicone barrier: 0.0, 2.0, 4.0, and 6.0 mm. The process of making composite resin specimens were as followed. 1) Placing the light-polymerized composite resin material into the lower compartment space between the 2 glass slabs 8.0 mm from bottom of the lower compartment. 2) Putting the silicone barrier with 2.0 mm thickness to the second experimental group (Si2), 4.0 mm thickness to the third experimental group (Si4), and 6.0 mm thickness to the fourth experimental group (Si6) on composite resin specimen in the middle compartment. 3) Polymerizing by a light- polymerizing unit for 40 seconds. 4) Removing 2 glass slabs and transferring the mold with composite resin specimen to a digital microhardness instrument. The output of light-polymerizing unit (600 mW/cm^2) was checked prior to each procedure by using radiometer. In brief, 4 groups of composite resin specimen were made. The first group (Si0) not using clear silicone barrier was a control group. The second (Si2), the third (Si4), and the fourth group (Si6) were test groups. The dimension of all composite resin specimens was 4 × 8 × 4 mm, and 40 specimens were made for each group. Therefore, 160 specimens were made for this study.

Investigating degree of conversion

1.1 The KHN of the composite resin

There were 4 experimental groups (Si0, Si2, Si4, and Si6) and 4 Subgroups (CD0, CD2, CD4, and CD6) in each experimental group. Four horizontal lines were drawed at 0.25 (CD0), 2.0 (CD2), 4.0 (CD4), and 6.0 (CD6) mm in depth from the top of composite resin specimen respectively. Measurement points were located at the center of horizontal center lines (Fig. 3). Thus, there were 16 subgroups in this study. For example, Si4CD2 represented 4.0 mm silicone barrier and was tested at 2.0 mm from the top of composite resin specimen. Furthermore, 10 specimens were made for each subgroup. Then, all specimens, which were still fixed in the mold, were transferred to a digital microhardness instrument and examined the Knoop hardness immediately (Fig. 4). An indenter load of 25 grams and the load holding time of 10 seconds were used. The specimens have been randomized with respect to the hardness testing. After that, the data were analyzed by Two-way ANOVA for all tests. The Tukey's test was used for multiple comparisons, with significance level ($p < 0.01$).

1.2 The percentage of bottom-top of KHN of the hardened composite resin

To characterize the polymerized conversion, the percentage of bottom-top of KHN of the deeper surface (2.0, 4.0, and 6.0 mm) against the irradiated top surface (0.25 mm) of hardened composite resins was determined in each experimental group. Therefore, there were 3 percentages of bottom-top KHN subgroup (subgroup PCD2-0, subgroup PCD4-0, and subgroup (subgroup PCD2-0, subgroup PCD4-0, and subgroup PCD6-0 respectively) in each experimental group. Thus, there were 12 subgroups in this study. Then, these percentages of bottom-top were analyzed by Two-way ANOVA for all tests. The Tukey's test was for multiple comparisons, with significance level ($p < 0.01$). In the present study, the only percentages of bottom-top of not less than 80 % were collected and represented as an appropriate polymerization.

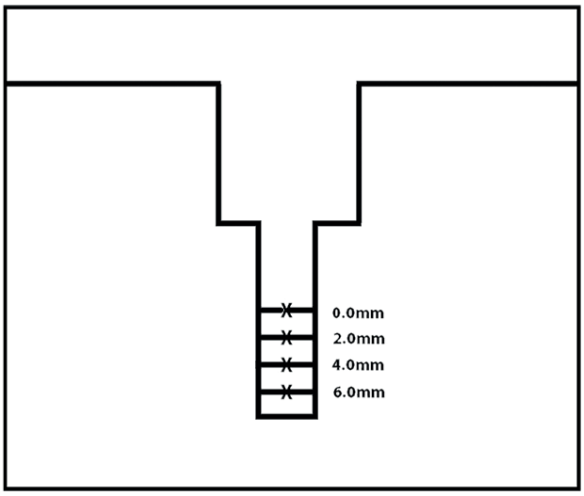

Figure 3 *The specimen is divided into four parts by underscore 3 horizontal lines separating each line 2 mm from the lower part of the L&P mold. Cross presents the indentation position.*

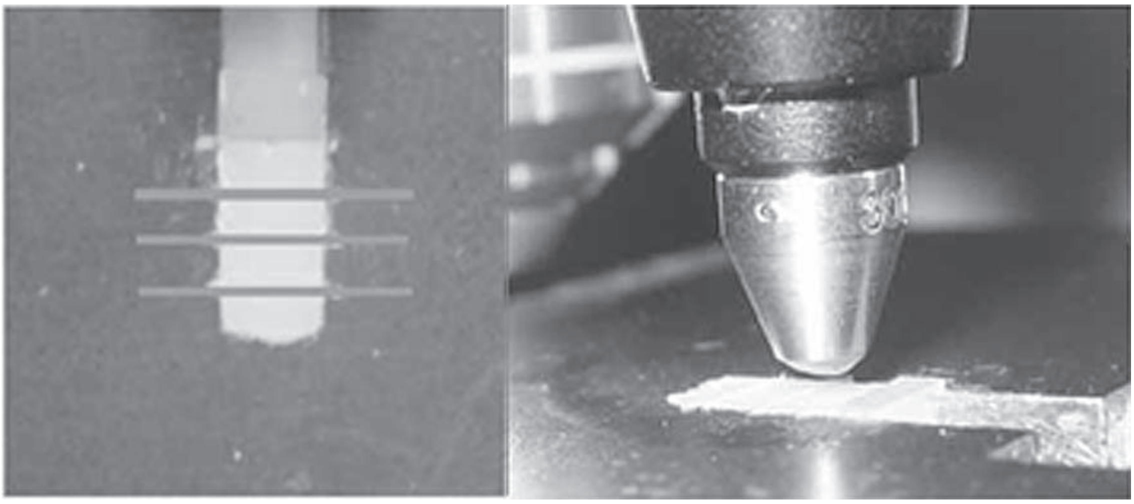

Figure 4 *Knoop Hardness Measurement*

Results

The mean KHN and their corresponding standard deviations of composite resin were investigated in term of a function of sample depth and clear silicone thickness. Then the data were summarized in Table 2. From the findings, Si0CD0 showed the maximum mean KHN (64.11), and there were significantly different ($p < 0.01$) among mean KHN in group Si0, Si2, Si4, and Si6, except Si6CD6. Besides, within each experimental group, there is an inverse proportion between the mean KHN and the depth of composite resin, except for Si6CD6. This is because composite resin material at Si6CD6 was too soft for an accurate indentation.

The mean percentages of bottom-top of KHN and their corresponding standard deviations of composite resin were investigated in term of a function of sample depth and clear silicone thickness. Then the data were summarized in Table 3. From the findings, Si4PCD2-0 showed the maximum percentage (92.11 %). Also there were significantly different ($p < 0.01$) among mean percentages of bottom-top of KHN in group Si0, Si2, Si4, and Si6, except Si6PCD6-0. In addition, within each experimental group, there is an inverse proportion between the mean percentages of bottom-top of KHN and the depth of composite resin, except for Si6PCD6-0. This is because composite resin material at Si6PCD6-0 was not hard enough for an accurate indentation. From the percentage of bottom-top of hardness (using 2.0, 4.0, and 6.0 mm as the bottom points and 0.25 mm as the top point), there were only 3 data which were not less than 80 %. They were 85.16 %, 85.64 %, and 92.11 % (using 2.0 mm as the bottom point and 0.25 mm as the top point) in Subgroup Si0PCD2-0, Subgroup Si2PCD2-0, and Subgroup Si4PCD2-0, respectively.

Table 2 Mean KHN and standard deviation of depth of light-polymerizing composite resin group investigated as function of clear silicone thickness group.

Depth of composite resin group	Si0 Mean KHN	Si0 SD	Si2 Mean KHN	Si2 SD	Si4 Mean KHN	Si4 SD	Si6 Mean KHN	Si6 SD
	Thickness of clear silicone group †							
CD0	64.11[a]	(0.52)	60.01[b]	(0.69)	52.01[d]	(0.79)	48.01[ef]	(1.00)
CD2	54.59[c]	(0.84)	51.39[d]	(1.11)	47.90[ef]	(1.12)	32.58[i]	(1.63)
CD4	49.50[e]	(0.91)	40.50[g]	(1.04)	35.48[h]	(0.99)	18.51[l]	(1.00)
CD6	47.51[f]	(1.04)	30.11[j]	(1.03)	25.03[k]	(0.99)	ND	

† Mean values and their standard deviations followed by the same letter in a column are not significantly different ($p < 0.01$).
ND = Not determined, because the material was too soft for an accurate indentation.

Discussion

The null hypotheses were rejected due to statistically significant differences among all groups. Therefore, the clear silicone thickness and the polymerizing depth of light-polymerized composite resin have direct effects on (1) the KHN of the composite resin and (2) the percentage of bottom-top of KHN of composite resin. In the present study, it was found that the mean KHN in each group differs significantly. In addition, the maximum KHN is 64.11 (Si0CD0). The increasing thickness of clear silicone reduced the mean of KHN. Moreover, the deeper the light-polymerized composite resin is, the less mean of KHN is shown. Consequently, it can be summarized that the thickness of clear silicone and the depth of light-polymerized composite resin are essential factors for incomplete polymerization. Degree of polymerization is one of the important factors that affect the mechanical properties and a clinical performance of composite resins.[10-12,23,44-51] Generally, KHN has been demonstrated as a good predictor of the effect of different light sources,[52] and a previously published study showed a significant correlation between the hardness of composite resin and degree of conversion.[53] Furthermore, the hardness testing is also a reliable and commonly-used method to test how well a composite resin is polymerized.[31] Therefore, KHN was used in the present study to reflect monomer conversion at different depths of tested light-polymerized composite resin.

Table 3 *Mean of percentage difference between bottom and top KHN value and standard deviation of the depth of light-polymerizing composite resin group investigated as function of clear silicone thickness group.*

	Thickness of clear silicone group †							
Percentage of bottom	Si0		Si2		Si4		Si6	
-top KHN group	Mean	SD	Mean	SD	Mean	SD	Mean	SD
PCD2-0	85.16[a]	(1.36)	85.64[a]	(2.34)	92.11[b]	(2.08)	67.86[c]	(3.31)
PCD4-0	77.22[d]	(1.46)	67.48[c]	(1.08)	68.20[c]	(1.42)	38.53[e]	(1.51)
PCD6-0	74.12[t]	(1.63)	50.15[g]	(1.19)	48.12[g]	(1.55)	ND	

† Mean values and their standard deviations followed by the same letter in a column are not significantly different ($p < 0.01$).
ND = Not determined, because the material was too soft for an accurate indentation.

When using the depth of polymerization based on top and bottom hardness measurements,[33-36] the results revealed that there were only 3 data in this study with the percentage of over 80 %. They were 85.16 %, 85.64 %, and 92.11 % in Subgroup Si0PCD2-0, Subgroup Si2PCD2-0, and Subgroup Si4PCD2-0, respectively (using 2.0 mm as the bottom point and 0.25 mm as the top point). Therefore, it can be concluded that light-polymerized composite resin of 2.0 mm thickness should be used, while the thickness of clear silicone is not more than 4.0 mm.

Distances from tip of the light-polymerizing unit to the top surface of light-polymerized composite resin could have an important effect on mechanical properties of composite resin.[5-8,18] The performance of the dental light-polymerizing unit should not be tested at 0.0 mm from the end of the light guide, instead, they should be tested at more clinically relevant distances. The previous studies have used 4 mm[54] or 5 mm[55] to represent an average distance, and 8 mm[54] or 9 mm[17,56] to represent an extreme situation. Moreover, previous research has shown that the distance of light tip to the gingival floor of a typical Class II preparation may be 7 mm or more.[57-59] Thus, in this study, the distance was 6.0 mm and represented a clinical situation by using L&P mold and method. By the way, the further study should determine the relation of Knoop Hardness Number and the different distance from tip of light-polymerizing unit to the top surface of the composite resin.

Thickness of index used for a direct and direct-indirect techniques could have an effect on mechanical properties of composite resin. However, there was no previous study. Hence, this research was designed to use silicone index which varied in thickness (2.0, 4.0, and 6.0 mm). From the result of this research, the index should have the thickness in range from 2.0 to 4.0 mm. Moreover, this seems to be practical in clinical application. If the index thickness is lower than 2.0 mm, the index tended to be distorted during clinical application. The distortion is a result of contour and anatomy of restored tooth. If the index thickness is higher than 4.0 mm, light-polymerized composite resin is not adequate for polymerization and tends to have lower microhardness and mechanical properties. Because of inadequate polymerization, the light-polymerized composite resin may be dislodged or distorted when the operator removes index from the restored tooth. By the way, the further study should determine the relation of the thickness of silicone index and distortion of silicone index.

Within the limitations of L&P mold and method, the indentations were determined at the side of specimens only. An advantage of measuring at the side of specimens is using fewer specimens. In addition, some research used various levels from top surface to prevent shape distortion of the indentation.[37-40] For example, Fe Silva used the level of 0.1 mm, not 0.0 mm.[38] Several researchers tended to prevent shape distortion of the indentation.[37-40] Hence, KHN could not be actually measured at 0.0 mm depth from the top of composite resin specimen, and 0.25 mm depth from the top of composite resin specimen was the minimum depth that KHN could be measured precisely. Therefore, the depth of composite resin 0.0 mm in Table 2 was actually represented by 0.25 mm. Moreover, the mean KHN using the level of 0.25 mm showed an appropriate result (64.11) which was not different from those who used 0.0 mm.[39,52,60] Hence, the depth of composite resin 0.25 mm corresponded with the mean KHN of the irradiated top surface.

Focusing on the percentages of bottom-top of hardness which were not less than 80 %, Figure 5 showed that slopes of line CD0 and line CD2 tended to have the similar pattern. While line CD0 was defined as a top point, line CD2 was defined as a bottom point. Moreover, the hardness measurements for the Si4 group were significantly lower than the Si0 and Si2 groups. The means KHN of Si4CD0 and Si4CD2 were lower than those of Si0 and Si2, but the mean KHN of Si4CD0 was closed to Si4CD2. It could be clarified that Si4PCD2-0 (92.11 %) was higher than Si0PCD0-0 (85.11 %) and Si2PCD2-0 (85.64 %). From the result of this study, the further study should determine the relation of duration of polymerizing light exposure and mean KHN of light-polymerized composite resin investigated in term of function of specimen depth and clear silicone thickness.

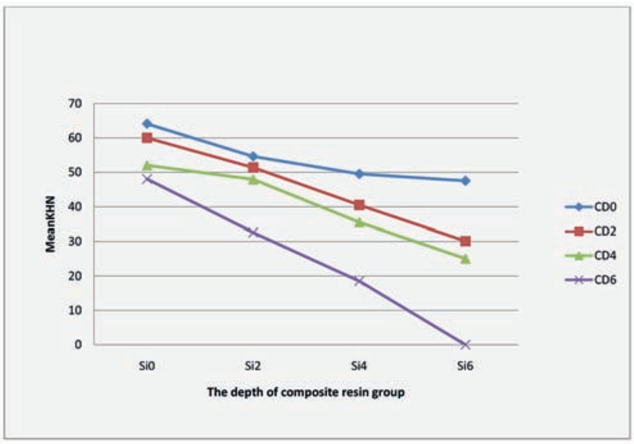

Figure 5 *Mean KHN of depth of light-polymerizing composite resin group investigated as function of clear silicone thickness group*

Conclusion

Under researchers' recommendation, the light-polymerize composite resin with no more than 2.0 mm of depth incorporated with the clear silicone barrier of no more than 4.0 mm thickness should be used, which showed the percentage of bottom-top hardness of no less than 80 %.

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