Original Study

# ASSESSMENT OF TOP AND BOTTOM SURFACE OF SILORANCE AND METHACRYLATE COMPOSITES BY USING VICKERS HARDNESS TESTING

*Davari A,[1] Amini M,[2] Daneshkazemi A[3]*

*1. Professor, Department of Cosmetic & Restorative Dentistry, School of Dentistry, Yazd University of Medical Sciences, Yazd, Iran*
*2. Dental General Practitioner, Department of Cosmetic & Restorative Dentistry, Yazd Dental School, Yazd University of Medical Sciences, Yazd, Iran*
*3. Associate Professor, Department of Cosmetic & Restorative Dentistry, School of Dentistry, Yazd University of Medical Sciences, Yazd, Iran*

**ABSTRACT**

**Aim:** The aim of this study was to compare the hardness of surface and depth of cure of methacrylate-based and silorane-based composites after curing with LED.

**Materials and Methods:** Forty composite discs were prepared using plastic molds with the inner diameter of 8 mm and a height of 2 mm. These samples were divided into two groups: Group A (P90) and Group B (Z250) which were cured and prepared. Then, the Vickers micro-hardness was measured on all samples from the superior surface close to the light source and the inferior surface. Finally, the gleaned data were coded and analyzed using descriptive statistics including mean, SD, independent T-test, and paired T-test. The level of statistical significance was set at p-value = 0.05.

**Results:** The mean VHN was higher in both superior and inferior surfaces of methacrylate-based composites compared to silorane-based composites ($p < 0.001$ and $p < 0.05$ respectively). There was a statistically significant difference in the mean VHN of superior and inferior surfaces in the silorane group ($p < 0.001$). There was not any statistically significant difference between the mean VHN of the inferior and superior surfaces of Z 250 composite ($p < 0.05$).

**Conclusions:** The curing depth of methacrylate composites exceeds far the curing depth of the silorane-based composites.

**Key Words:** Curing depth; Methacrylate-based composites; Silorane-based composites; Vickers hardness test.

## Introduction

Composites are composed of three main constituents: organic matrix, non-organic filling components, and the binding factor or silane.[1] The resin matrix is the active chemical component of composites and the polymeric matrix is formed during a radical increment polymerization reaction between oligomers.[2] The scattered non-organic components include constituents such as glass or fine quartz or the microfine colloidal silica. The inclusion of these components to the resin matrix reduces polymerization contraction, improves mechanical properties like strength and hardness, creates radiopacity, decreases the thermal expansion index of resin matrices, and controls the aesthetic appearance of composites such as color and translucency. Silica-based fillers are hydrophilic because there is a layer of hydroxyl groups on their surface. The binding substance is silane which has functional groups like methoxy at its two ends that is hydrolyzed. $RSi(OMe)_3$ is hydrolyzed to $RSi(OH)_3$ and interacts with OH filler group (substrate$(OH)_3$. It is bonded to the filler surface. On the other hand, it has methacrylate group on its other end which is bonded to the matrix via the dual bond of carbon.[3]

Unlike the methacrylate-based composites in which the radicals trigger the initiation of polymerization reaction, the siloranes are a cationic monohybrid system with expanding cycles fabricated to overcome the problems induced by polymerization contraction.[4] These monomers are produced by the reaction between oxyrane and cycloxan molecules, i.e., a process which is not sensitive to oxygen. This hinders the formation of oxygen inhibition layer at the composite surface. The formation of this layer is one of the shortcomings of methacrylate-based composites. It is formed by the inhibition of primer radicals by oxygen. Compared to methacrylate, silorane enjoys less volume contraction, less contraction stress, higher flexional strength, higher resistance to fracture, higher color stability, absence of cellular toxicity, better biocompatibility, insolubility in water, and lower translucency.[5]

The light emitting diodes (LEDs) have been used for composite curing for the first time in 1995. LEDs emit a specific wavelength 470 nm to activate camphoroquinone. These lamps work for 10000 h and provide a high energy performance. They consume little power and can be battery-operated.[6]

The degree of composite conversion affects its mechanical properties, solubility, stability of dimensions, color change, and biocompatibility.[7] The curing depth and micro hardness test are reliable features used widely in assessing the degree of resin conversion, and consequently, in employing the light sources.[8] Hardness is greatly dependent on composite type,[9] polishing,[10] maintenance conditions[11] and curing conditions like distance from the light source, intensity, and duration of time.[12] The effect of depth of curing on the microhardness in different composites have been evaluated in a limited number of studies.[12-13] The present in vitro study assessed microhardness of surface and depth of cure on methacrylate-based and silorane-based composites after LED curing.

## Materials and Methods

In this study, we used Filtek Z250 (3M ESPE, St Paul, USA) and Filtek P90 (3M ESPE, St Paul, USA). The characteristics of the restorative materials used in this study are given in Table 1. A total of 40 composite discs were prepared using plastic molds with inner diameter of 8 mm and height of 2 mm and divided into Group A (P90) and Group B (Z250). The molds were placed on glass slabs. Then, the P90 and Z250 composites were invested using the mass method. A celluloid film was put on it and another

glass slab was pressed on it to remove the overflow composite so that a composite thickness of 2 mm was achieved. Subsequently, the height of the samples was cured for 20 s from a zero distance (the tip of the light cure device was completely attached to composite surface) with LED device (Dentamerica LITEX 696 CORDLESS LED Curing Light) on the basis of the manufacturer's instructions using the standard method at 1200 mw/cm$^2$ intensity. Also, a black light-absorbing plate was placed under the device to prevent light reflection. Then, the samples were separated from the mold and their surfaces were polished and finished. The upper surfaces of the samples were marked with an indelible mark and the samples were placed in dark room for 24 h at room temperature. Next, the samples were taken out of the molds and kept in metallic dishes containing distilled water till the time of preparation of other samples. Twenty samples were prepared from each of A(P90) and B(Z250) composites, so, there were a total of 40 composite samples placed within a container of 20 mL of distilled water and then dried. Vickers Hardness Test was performed on all samples on the surface close to the light source and on the inferior surface opposite to it using Vickers Hardness testing Machine (Copa MHI, Iran) with a force of 50 g for 15 s. The results were gleaned for statistical analysis.

| Materials | Organic Matrix | Nonorganic Filler | Filler vol./wt. percentage | Filler Size |
|---|---|---|---|---|
| Filtek P90 3M.ESPE, st. Paul, USA | Silorane | Quartz, Yttrium Fluoride | 76/55 | 0.47 µm |
| Filtek Z250 3M.ESPE, st. Paul, USA | Bic GMA,UEDMA, Bis-EMA | Zirconia, Silica | 84.5/60 | 0.01-3.5 µm |

*Table 1: Composites Characteristics*

The degree of hardness was measured with Vickers hardness testing device (Copa MHI, Iran) and recorded in a special data collection form designed for this purpose. The gleaned data were coded, imported to SPSS18 and analyzed using descriptive statistics including mean, SD, independent T-test, and paired T-test. The level of statistical significance was set at P-value=0.05.

**Results**

The mean and standard deviation related to the experimental materials are presented in Table 2 and 3. The mean VHN of both the inferior and superior surfaces of methacrylate-based composites is significantly higher than silorane-based composites (p=0.0001) (p=0.020). In the P90 composite, the cure depth of the superior surface was statistically significantly higher than the inferior surface (P-value=0.0001), yet, the mean difference in cure depth of the inferior and superior surfaces of Z250 composite is not statistically significant (P-value=0.120).

| Groups | Surface | N | Mean | SD | p Value |
|---|---|---|---|---|---|
| Group A (P90) | Top | 20 | 79.80 | 4 | 0.0001 |
| Group B (Z250) | Top | 20 | 86.86 | 5.8 | |
| Group A (P90) | Bottom | 20 | 77.57 | 3.9 | 0.020 |
| Group B (Z250) | Bottom | 20 | 84.03 | 5.6 | |

*Table 2: Results of statistical analysis of hardness in two composite groups compared.*

| Groups | Surface | N | Mean | SD | p Value |
|---|---|---|---|---|---|
| Group A (P90) | Top | 20 | 79.80 | 4 | 0.0001 |
| | Bottom | 20 | 77.57 | 3.9 | |
| Group B (Z250) | Top | 20 | 86.86 | 5.8 | 0.120 |
| | Bottom | 20 | 84.03 | 5.6 | |

*Table 3: Results of statistical analysis of both the inferior and superior surfaces in each group*

**Discussion**

This study compared the hardness of the inferior and superior surfaces of silorane-based and methacrylate-based composites. The study was conducted on P90 as a silorane-based composite and Z250 as a methacrylate-based composite. The Vickers harness test indicated that the hardness value of silorane-based composite was smaller than the hardness value of acrylate-based composite. The difference may be attributed to the chemical differences of monomers,[12] and type and scattering of nonorganic constituents.[14] The study by de Moreas Porto *et al.*[15] used the near-IR spectroscopy to measure the DC (degree of conversion) of composites. This method uses the difference in intensity of absorption and reflection of light by composite in the cured and non-cured states. The results demonstrated the greater degree of conversion (DC) of methacrylate-based composite which can lead to greater microhardnesss of Z250 composite. Also, in the study by Son *et al.*[14], although the DC of P90 composite was higher than the DC of Z250 composite, the Vickers hardness (HV) of Z250 composite was much greater than the Vickers hardness of P90 composite. It is said that the hardness value is not always a predictor of DC and the higher H of Z250 composite is attributed to higher percentage of composite filler, so that the less the filler and the more the monomer, the smaller the HV.

One reason for the difference in hardness of composites can be attributed to composite heat loss when it is removed out of the carpool till it is transmitted to the mold. It is reported that the composite temperature may fall by 50% in 2 min during the time it is taken out of the carpool depending on the type and brand of the composite.[16]

According to the study by Gonzalez,[17] the P90 composite has the highest hardness at 700 mW/cm$^2$. Also, according to da Silva *et al.*[18], the micro hardness value of conventional composites increases at higher intensities of

the light cure device. Some scholars believe that the increased emission intensity of light cure device may produce more heat in the composite leading to increased kinetic motion of monomers which results in promoted DC of composites and subsequently, induces increased micro hardness. In this study, the emission of 1200 mW/cm$^2$ in the LED device was stable on the basis of the manufacturer's instructions.

In the study by Mohammadi *et al*.[19], the researchers investigated the effect of pre-heating on the mechanical properties of P90 and Z250 composites. They placed the composites in a container with 25, 37 and 68° C water for 15 min and then invested them in molds and cured them. The flexional strength and the elastic module were measured using three-point bending test and the surface strength was measured with Vickers hardness test. The results showed that pre-heating leads to increased elastic module and micro hardness, yet it had no significant effect on flexional strength. Also, Z250 showed higher results in all tests compared to P90 so that increased temperature of pre-heating had led to greater micro hardness of Z250 compared to P90 composite. Hence, it could be explained that if the emission intensity of the LED device is increased, a higher temperature is produced which directly affects the mechanical properties of the composites, specially the Z250.

Cellabus *et al*.[7] investigated the effect of exposure time of LED and halogen sources on the hardness of composites. They found that increasing the exposure time from 20 s to 40 s increased the composite hardness on all surfaces specially in higher depths, albeit, of course, our study emphasized 20 s exposure time for depths less than 2.5 mm. Furthermore, Cardia *et al*. (2015) investigated the cure depth of silorane-based and Z250 composites and found that the difference in composite hardness with exposure times of 20 s and 40 s of LED exposure was not significant.

After curing, the samples were polished with finishing discs. The study by Kaminedi *et al*.[20] was carried out to investigate the effect of finishing and polishing on the physical properties of Z250. In this study, immediate finishing after curing caused the highest micro hardness for methacrylate-based composite. Grinding and polishing of the samples are the prerequisites of any hardness test using any method with any device. If the site of hardness testing is not properly polished, the obtained hardness value will be under-reported due to projections, porosities, and roughness and the created depression will not be readily pronounced and legible.

Moreover, the study by Kusgoz *et al*.[11] investigated the four properties of cure depth, surface hardness, DC, and cervical microleakage as important clinical parameters on silorane-based and methacrylate-based composites during the three 7-day, 1-day, and 30-day periods. The DC of methacrylate-based composite was greater than the DC of silorane-based composite and the disparity is attributed to the difference in the structure of monomers and fillers. Increasing the immersion time of the samples from 1 day to 30 days resulted in increased DC of the samples, however, the hardness of the superior and inferior surfaces of the composites except silorane decreased. The difference in DC and KHN in the course of time is attributed to insufficiency of only DC in the three-dimensional structure of composites, unequal content of C=C in various regions of the composite and the presence of unreacted monomers besides the reacted monomers. Additionally, the immersion of composites in water reduces KHN in the course of time due to the absorption of water by the resin component, matrix inflation, and reduced force of polymeric chains. On the other hand, maintenance of silorane-based composite in water has no effect on KHN. This may be attributed to the structure of silorane and reduced water absorption and their solubility against methacrylate.

de Moraes *et al.* [21] studied the effect of 6-month immersion of microhybrid composite in water on surface hardness. This study used Z250 composite. As predicted, after 6 months of maintenance in a dark chamber filled with distilled water, the hardness of the superior surface of the composite was reduced to less than the hardness in the first 24 h due to resin matrix inflation, reduced cohesion between the polymer chains, and also reduced cohesion between the filler and the resin. Nevertheless, contrary to our expectation, the hardness of the inferior surface was greater than the hardness of the superior surface of the composite. It was explained that the free radicals that are the primers (initiators) of the reaction in methacrylate-based composites, are surrounded by the components of the reaction from three dimensions in the major volume of the composite while they are surrounded from just one dimension in the superior surface. On the other hand, increasing the temperature (photo activation) during light cure is greater in the deeper layers of the composite than the superficial layers since heat transmission is smaller. It has been proved that even minor increase in temperature leads to increased hardness.

## Conclusions

Based on the results of the present study, the hardness of methacrylate-based composites (Z250) was higher in both the superior and inferior surfaces compared to silorane-based composite (P90). Also, the hardness of the superior surface (close to the light source) was higher than the inferior surface (away from the light source) for P90 composite.

## Acknowledgements

This article is based on the thesis by Mohammadreza Amini which supervised by Dr Davari and Dr Daneshkazemi in Yazd Dental School. I would like to express his gratitude towards them for their guide and help during the research. The authors also thank Arefeh Dehghani tafti Statistician in Yazd University of Medical Science for hers help with the statistical analysis.

**References**


1. Krifka S, Federlin M, Hiller KA, Schmalz G. Microleakage of silorane-and methacrylate-based class V composite restorations. Clin Oral Investig 2012;16(4):1117-24.
2. van Noort R. Introduction to dental materials. 3rd ed2007. 316 p.
3. Powers JM, Sakaguchi RL. Craig's Restorative Dental Materials. 12th ed: Mosby Elsevier; 2006. 632 p.
4. Al-Boni R, Raja OM. Microleakage evaluation of silorane based composite versus methacrylate based composite. J Conserv Dent 2010;13(3):152-5.
5. Arocha MA, Mayoral JR, Lefever D, Mercade M, Basilio J, Roig M. Color stability of siloranes versus methacrylate-based composites after immersion in staining solutions. Clin Oral Investig 2013;17(6):1481-7.
6. Malhotra N, Mala K. Light-curing considerations for resin-based composite materials: A review. Part II. Compend Contin Educ Dent 2010;31(8):584-8.
7. Ceballos L, Fuentes MV, Tafalla H, Martínez Á, Flores J, Rodríguez J. Curing effectiveness of resin composites at different exposure times using LED and halogen units. Med Oral Patol Oral Cir Bucal 2009;14(1):E51-6.
8. Price RB, Fahey J, Felix CM. Knoop microhardness mapping used to compare the efficacy of LED, QTH and PAC curing lights. Oper Dent 2010;35(1):58-68.
9. Rueggeberg FA, Jordan DM. Effect of light-tip distance on polymerization of resin composite. Int J Prosthodont 1993;6(4):364-70.
10. Davari AR, Kazemi AD, Ataei E, Vatanpour M, Abdollahi H. Effects of bleaching and remineralizing agents on the surface hardness of enamel. J Dent. 2012;13(4):156-63.
11. Kusgoz A, Ülker M, Yesilyurt C, Yoldas OH, Ozil M, Tanriver M. Silorane-Based Composite: Depth of Cure, Surface Hardness, Degree of Conversion, and Cervical Microleakage in Class II Cavities. J Esthet Restor Dent 2011;23(5):324-35.
12. Cardia GS, Ishikiriama SK, Borges AFS, Bombonatti JFS, Furuse AY, Francisconi PAS. Curing depth of a silorane-based resin composite. Revista Sul-Brasileira de Odontologia. 2015;12(2):196-201.
13. García-Contreras R, Scougall-Vilchis R, Acosta-Torres L, Arenas-Arrocena C, García-Garduño R, de la Fuente-Hernández J. Vickers microhardness comparison of 4 composite resins with different types of filler. J Oral Res 2015;4(5):313-20.
14. Son S-A, Roh H-M, Hur B, Kwon Y-H, Park J-K. The effect of resin thickness on polymerization characteristics of silorane-based composite resin. Restor Dent Endod 2014;39(4):310-8.
15. Porto IC, de Aguiar FH, Brandt WC, Liporoni PC. Mechanical and physical properties of silorane and methacrylate-based composites. J Dent 2013;41(8):732-9.
16. Nada K, El-Mowafy O. Effect of precuring warming on mechanical properties of restorative composites. Int J Dent 2011;2011:536212.
17. Gonzalez M, Poskus L, Sampaio Filho H, Perez CR. Influence of irradiance and exposure time on the degree of conversion and mechanical properties of a conventional and silorane composite. Indian J Dent Res 2013;24(6):719-22.
18. da Silva EM, Poskus LT, Guimarães JG. Influence of light-polymerization modes on the degree of conversion and mechanical properties of resin composites: a comparative analysis between a hybrid and a nanofilled composite. Oper Dent 2008;33(3):287-93.
19. Mohammadi N, Jafari-Navimipour E, Kimyai S, Ajami A-A, Bahari M, Ansarin M *et al*. Effect of pre-heating on the mechanical properties of silorane-based and methacrylate-based composites. J Clin Exp Dent 2016;8(4):e373-e378.
20. Kaminedi RR, Penumatsa NV, Priya T, Baroudi K. The influence of finishing/polishing time and cooling system on surface roughness and microhardness of two different types of composite resin restorations. J Int Soc Prev Community Dent 2014;4(Suppl 2):S99-S104.
21. de Moraes RR, Marimon JL, Schneider LF, Sinhoreti MA, Correr-Sobrinho L, Bueno M. Effects of 6 months of aging in water on hardness and surface roughness of two microhybrid dental composites. J Prosthodont. 2008;17(4):323-6.


**Corresponding Author**


Dr. Mohammadreza Amini
Dental General Practitioner,
Department of Cosmetic & Restorative Dentistry,
Yazd University of Medical Sciences,
Yazd, IRAN
Email Id: - biology_2011@yahoo.com