Evaluation of surface hardness and curing depth of two different light curing composite resins system: An in vitro study

Dr. Pragya Patel, Dr. Sanjeev Tyagi, Dr. Abhishek Bajolia, Dr. Neha Bedwal, Dr. Manoj Jain and Dr. Ankita Bhargava

DOI: https://doi.org/10.22271/oral.2021.v7.i3b.1286

Abstract
This in-vitro study was done to compare the effect of LED light curing system on polymerization and hardness of two composite resins system. A Total of 40 samples, 20 samples prepared using silorane based – Filtek LS and 20 samples prepared using methacrylate based- Filtek Z350 XT in a plastic molds with diameter of 8 mm and thickness of 3 mm and cured with LED curing light. knoop hardness testing was done using a 50grams load and a dwell time of 15sec and percentage depth of cure is calculated and statistical analysis was performed using Student t- test. Results showed that silorane based composite was found to have better hardness n depth of cure than methacrylate based composite. Top surface have better hardness than bottom surface.

Keywords: surface hardness, curing depth, composite resins system

1. Introduction
The development of composite materials began in the ‘40s of the twentieth century in Germany by synthesizing a molecule of PMMA (polymethyl methacrylate). In the ‘50s, research works began on the creation of composite materials by adding inorganic fillers. In 1955 the phenomenon of increase in adhesion was discovered by using enamel etching with acid, which allowed the initiation of the development of so called “adhesive dentistry”. The breakthrough was to replace the resin based on PMMA by synthesized by Bowen and Cobb bis-GMA resin. [1, 2]

To overcome the problems associated with polymerization shrinkage, early attempts focused on the type and amount of the particles included in composite resins and on different applications to particle surfaces. Later studies focused on the relationship between the polymerization shrinkage and the monomers composing the organic matrix of composite resins. For this reason, the 3M-ESPE Company developed the silorane matrix system, which differs from methacrylate-based monomers and released the first composite filler material in which this matrix system was used: Filtek Silorane. Silorane actually comprises two different monomers called siloxane and oxirane [4].

The selection of an efficient light curing unit (LCU) is critical factor for the bonded resin restorations. Since, the LCU should provide adequate degree of conversion for both the adhesive and resin composite [5]. These new light sources including LED units have a light intensity up to 1200 mW/cm2. However, increasing the light intensity doesn’t mean that sufficient polymerization will be obtained [6].

Surface hardness is one of the important characteristics of composite resins which can affect the clinical success rate of restorations. Nowadays various types of composite resins are available based on different fillers, which provides different hardness of composite resins too. [7]

Depth of cure and microhardness testing have been reliably and widely used to assess the relative degree of cure of resins, and thus the efficiency of light sources.
The Knoop microhardness test has been shown to be one of the best indirect methods for testing the hardness of resin composite[10].

The present in vitro study was carried out to compare methacrylate-based and silorane –based composite resin based on surface hardness and curing depth by using LED curing unit.

2. Materials and Methods
Custom-made plastic molds with inner diameter of 8 mm and thickness of 3 mm were taken. Samples were prepared by placing molds on mylar strips which was placed on a glass slab and filled with composite material using composite instruments (GDC Titanium coating instrument) TNCIPCM#B, TNCFIS/M#B. Material were packed inside the mold cavity in two increments, 1st increment of 1.5mm was placed inside the mold. LED curing light (Bluephase) was used to cure the samples, with a light intensity of 1000mw/cm2 and cured for a period of 40 seconds, light was kept at a distance of 1mm from the mold.

After filling 2nd increment of 1.5mm another mylar strip was placed on upper surface and pressed with a glass slide to remove the excess material and flat the upper surface. Glass slide was removed, leaving mylar strip and light cure the material.

Samples were removed from the molds and polished and measured using micrometer. Top surface was marked with marker.

20 samples were prepared using Silorane based – Filtek LS A2 shade and 20 samples with methacrylate based- Filtek Z350 XT A2 shade. All 40 samples were kept in a dry at room temperature in lightproof container for 24hrs, and subjected for knoop hardness testing.

Samples were divided into two groups (n=20)

Group I (n=20) – silorane based composite resin (Filtek LS,3M ESPE)

Group II (n=20) – methacrylate based composite resin (Filtek 350 XT)

2.1 Knoop hardness testing
It was performed by using a 50grams load and a dwell time of 15sec. In each sample, three indentation were marked on top and bottom surface. The mean hardness value for both the surface will be noted and percentage depth of cure were calculated using:

Percentage depth of cure = bottom surface hardness / top surface hardness X 100

Statistical analysis was done using Student t- test and analyzed by Statistical Package of Social Science (SPSS Version 20; Chicago Inc., USA).

3. Results and Discussion

Table 1: Comparative analysis of the mean surface hardness (KHN) of two composite (Silorane & methacrylate) resins system on Top surface.

| Groups                        | Surface Hardness (KHN) on Top Surface |
|------------------------------|-------------------------------------|
|                              | MEAN | SD    | RANGE      |
| Group I [Silorane Based]     | 53.729 | 3.147 | 49.83-59.38 |
| Group II [Methacrylate]      | 43.836 | 2.059 | 40.05-47.89 |

Student ‘t’ Test Value
Significance ‘p’ Value
0.001(HS)

Table 1 reveals the Mean surface hardness on top surface was more in silorane based composite resin as compare to methacrylate based Composite Resin. There was statistically highly significant difference in mean surface hardness (KHN) of two composite (Silorane & methacrylate) resins system on Top surface. (p=0.001)

Table 2: Comparative analysis of the mean surface hardness (KHN) of two composite (Silorane & methacrylate) resins system on Bottom surface

| Groups                        | Surface Hardness (KHN) on Bottom Surface |
|------------------------------|-----------------------------------------|
|                              | MEAN | SD    | RANGE      |
| Group I [Silorane Based]     | 47.289 | 1.731 | 42.34-49.38 |
| Group II [Methacrylate]      | 37.439 | 1.328 | 35.09-40.09 |

Student ‘t’ Test Value
20.189
Significance ‘p’ Value
0.001(HS)

Table 2 reveals Mean surface hardness on bottom was more in silorane based composite resin as compare to methacrylate based Composite Resin. There was statistically highly significant difference in mean surface hardness (KHN) of two composite (Silorane & methacrylate) resins system on Bottom surface. (p=0.001)

Table 3: Comparative analysis of the mean% Depth of cure of two composite (Silorane & methacrylate) resins system.

| Groups                        | Mean% Depth of cure |
|------------------------------|---------------------|
|                              | MEAN | SD    | RANGE      |
| Group I [Silorane Based]     | 88.369 | 7.053 | 74.85-98.74 |
| Group II [Methacrylate]      | 85.640 | 5.964 | 75.19-95.13 |

Student ‘t’ Test Value
1.321
Significance ‘p’ Value
0.194(NS)

In both the Composite Resins system mean surface hardness was more on top surface as compare to bottom surface. There was statistically highly significant difference in mean surface hardness (KHN) between top & bottom surface among both the Composite Resins system (p=0.001)
Table 3 reveals Mean% Depth of cure was slightly more in silorane based composite resin as compare to methacrylate based Composite Resin. There was statistically no significant difference in mean% Depth of cure between two composite (Silotane & methacrylate) resins system. (p=0.194)

4. Discussion
The hardness of the composite is directly related to the degree of polymerization, and thus a good indicator of the degree of conversion of composite resins and a valuable parameter to estimate the mechanical properties. DeWald and Ferracane\textsuperscript{11} have stated that Knoop hardness correlates well with the degree of conversion. Also, it minimizes the effect of elastic recovery, is a relatively simple technique and show reliability of obtained result, hence it was the method chosen in this study.

Surface microhardness is considered as an indicative factor of the mechanical strength of a resin and correlates well to the material’s rigidity\textsuperscript{12}. In the current study, all test samples were cured on same parameter of light-curing method and slight finishing were done to remove soft resin layer material and to produce a relatively stable surface for testing. Hardness evaluation was used as an indirect method to verify the degree of conversion of composite resins\textsuperscript{13}.

In this in- vitro study Knoop hardness for silorane-based composite was higher than methacrylate-based composite. Moreover, composite hardness is influenced by several factors, such as organic matrix composition, type and amount of filler particles and degree of conversion\textsuperscript{14}. The organic matrix of Filtek LS is composed mainly by silorane resin and the inorganic particles are (76% by weight) in combination of fine quartz particles and radiopaque yttrium fluoride. In contrast, the organic matrix of Filtek Z350 XT is composed mainly by bis-GMA, UDMA, TEGDMA, and bis-EMA resins. To moderate the shrinkage, PEGDMA has substituted for a portion of the TEGDMA resin, and a combination of inorganic particles (72% by weight) of aggregated zirconia/silica cluster filler\textsuperscript{15}. For this reason, The higher Knoop hardness obtained for Filtek LS may be explained by differences in the filler type and organic matrix composition between the materials.

Silorane-based composite shows cationic polymerization reaction. It is characterized by continuous ring-opening expansion initiated at the time of curing and promoted further crosslinking and hardening of the entire matrix\textsuperscript{16, 17}. This cationic reaction is initiated by an acidic cation that allows stress relaxation, thereby, reducing polymerization contraction of the composite\textsuperscript{17, 18}. Silorane resin is composed mainly of siloxane and oxirane moieties\textsuperscript{18}. This new monomer is capable of being polymerized and continuing the cationic reaction in dark which is called self or dark polymerization\textsuperscript{19}. The dark reaction usually is time dependent and may attribute to the strength and hardness of the material\textsuperscript{20}. This might be the reason for silorane-based composite showed higher surface hardness value than methacrylate–based composite.

It has been reported that resin-based filling materials should exhibit a minimum of 80% bottom/top hardness percentage when cured in a 2-mm increment in order to be considered as adequately polymerized\textsuperscript{21}. So we have prepared the molds with 3mm depth and incremental filling was done so that proper polymerization take place.

The composite materials showed higher hardness values on the top surface than the base in all test groups. This can be explained by the higher degree of polymerization that occurs as a result of the closest contact of the light-curing guide to the top surface. When the curing light is applied to composite resin, some of the light rays are absorbed while others are scattered by the composite resulting in reduction or attenuation of light intensity which decreases the effectiveness of cure at the base surface\textsuperscript{22}.

However, it has been suggested that a composite resin specimen has been adequately cured when there is no more than a 20% difference between the maximum hardness at the top of the composite and the maximum hardness at its bottom\textsuperscript{23}.

So in our study both groups were exposed to same parameter of light curing, there was no significant difference in depth of cure between silorane –based composite and methacrylate-based composite.

5. Conclusions
Within the limitations of this in vitro study it can be concluded that
1. Surface hardness of silorane based composite was found to be better than methacrylate based composite.
2. Hardness on Top surface was found to more than bottom surface in both the groups.
3. Depth of cure was slightly higher in silorane based composite but there was no significant difference between the groups.

6. References
1. Powers JM, Sakaguchi RL. dental materials, H. Limanowska-Shaw, J. Shaw. Wroclaw, Urban & Partner, 2008.
2. Saghirí MA, Banava S, Sabzian MA et al. Correlation between long-term in vivo amalgam restorations and the presence of heavy elements in the dental pulp, Journal of Trace Elements in Medicine and Biology 2014;28(2):200-204.
3. Ilie N, Jelen E, Clementino-Luedemann T, Hickel R. Low-shrinkage composite for dental application. Dent Mater J 2007;26:149-55. [PubMed]
4. Shenoy A. Is it the end of the road for dental amalgam? A critical review. J Conserv Dent 2008;11:99-107.[PubMed]
5. Souza-Junior EJ, Araújo CTP, Prieto LT, Paulillo LAMS. Influence of the LED curing source and selective enamel etching on dentin bond strength of self-etch adhesives in class I composite restorations. Lasers Med Sci. 2012;27:1175-1182.
6. Rode KM, Kawano Y, Turbino ML. Evaluation of curing light distance on resin composite microhardness and polymerization. Oper Dent 2007;32(6):571-578.
7. Friedman J. Care and maintenance of dental curing lights. Dent Today. 1991;10(1):401. [PubMed]
8. Rode KM, Kawano Y, Turbino ML. Evaluation of curing light distance on resin composite microhardness and polymerization. Oper Dent 2007;32(6):571-578. [PubMed]
9. Aguiar FH, Andrade KR, Leite Lima DA, Ambrosano GM, Lovadino Jr. Influence of light curing and sample thickness on microhardness of a composite resin. Clin Cosmet Investig Dent 2009;1:21-5.
10. Price RB, Fahey J, Felix CM. Knoop microhardness mapping used to compare the efficiency of LED, QTH and PAC curing lights. Oper Dent 2010;35:58-68. [PubMed]
11. DeWald JP, Ferracane JL. A comparison of four modes of evaluating depth of cure of light activated...
composites. J Dent Res, 1987;66:727-30. [PubMed]
12. Eldiwany M, Powers JM, George LA. Mechanical properties of direct and post-cured composites. American Journal of Dentistry 1993;6(5):222-4. [Links]
13. Ferracane JL. Correlation between hardness and degree conversion during the setting reaction of unfilled dental restorative resins. Dent Mater 1985;1:11-14. [Links]
14. Correr AB, Sinhoreti MA, Sobrinho LC, Tango RN, Schneider LF, Consani S. Effect of the increase of energy density on Knoop hardness of dental composites light-cured by conventional QTH, LED and xenon plasma arc. Braz Dent J 2005;16:218-224.
15. Filtek Z350 XT Technical Product Profile - 3M multimedia.3m.com/mws/media/.../filtek-z350-xt-technical-product-profile.pdf
16. Palin WM, Fleming GJ, Burke FJ, Marquis PM and Randall RC. Monomer conversion versus flexure strength of a novel dental composite. Journal of Dentistry. 2003; 31(5):341-51. [Links]
17. Palin WM, Fleming GJ, Burke FJ, Marquis PM and Randall RC. The influence of short and medium-term water immersion on the hydrolytic stability of novel low-shrink dental composites. Dental Materials. 2005; 21(9):852-63. [Links]
18. Guggenberger R and Weinmann W. Exploring beyond methacrylates. American Journal of Dentistry. 2000; 13(Spec No):82D-4D. [Links]
19. Decker C, Viet C, Thi H. Photoinitiated cationic polymerization of epoxides. Polymer International 2001;50(9):986-97. [Links]
20. Oréfice RL, Discacciati JAC, Neves AD, Mansur HS, WCJ. Material Behaviour In situ evaluation of the polymerization kinetics and corresponding evolution of the mechanical properties of dental composites. Polymer Testing 2003;22(1):77-81. [Links]
21. Bouschlicher MR, Rueggeberg FA, Wilson BM. Correlation of bottom-to-top surface microhardness and conversion ratios for a variety of resin composite compositions Operative Dentistry 2004;29(6):698-704. [Medline]
22. Lindberg A, Peutzfeldt A, van Diik JW. Effect of power density of curing unit, exposure duration, and light guide distance on composite depth of cure. Clinical Oral Investigations 2005;9(2):71-6. [Links]
23. Dunn WJ, Bush AC. A comparison of polymerization by light-emitting diode and halogen-based light-curing units. J Am Dent Assoc 2002;133:335-341. [Links]