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

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#### 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 and 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. <sup>3</sup> 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/cm<sup>2</sup>. 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. <sup>7</sup> The distance of composite resins from the light curing source is another factor that influences the hardness. It is proved that the hardness is higher in lower curing distances [8, 9].

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, 1<sup>st</sup> 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/cm<sup>2</sup> and cured for a period of 40 seconds, light was kept at a distance of 1mm from the mold.

After filling 2<sup>nd</sup> 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=40)

**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 Composite Resin]	53.729	3.147	49.83-59.38
Group II [Methacrylate Based Composite Resin]	43.836	2.059	40.05-47.89
Student 't' Test Value	11.761		
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 Composite Resin]	47.289	1.731	42.34-49.38
Group II [Methacrylate Based Composite Resin]	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)

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:** 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 Composite Resin]	88.369	7.053	74.85-98.74
Group II [Methacrylate Based Composite Resin]	85.640	5.964	75.19-95.13
Student 't' Test Value	1.321		
Significance 'p' Value	0.194(NS)		

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 (Silorane & 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<sup>11</sup> 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<sup>[12]</sup>. 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<sup>[13]</sup>.

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<sup>[14]</sup>. 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<sup>[15]</sup>. 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<sup>[16, 17]</sup>. This cationic reaction is initiated by an acidic cation that allows stress relaxation, thereby, reducing polymerization contraction of the composite<sup>[17, 18]</sup>. Silorane resin is composed mainly of siloxane and oxirane moieties<sup>[18]</sup>. This new monomer is capable of being polymerized and continuing the cationic reaction in dark which is called self or dark polymerization<sup>[19]</sup>. The dark reaction usually is time dependent and may attribute to the strength and hardness of the material<sup>[20]</sup>. 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<sup>[21]</sup>. 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<sup>[22]</sup>.

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<sup>[23]</sup>.

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.

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